

**ANALYSIS AND MEASUREMENT OF THERMOPHYSICAL
PROPERTIES BY TEMPERATURE OSCILLATION**

**A THESIS SUBMITTED IN FULFILLMENT OF
THE REQUIREMENT FOR THE DEGREE**

OF

Doctor of philosophy

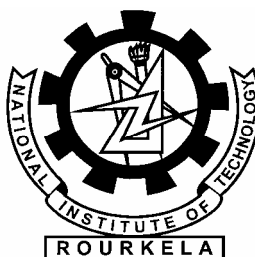
IN

MECHANICAL ENGINEERING

BY

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CERTIFICATE

This is to certify that the thesis entitled “**Analysis and Measurement of Thermophysical Properties by Temperature Oscillation**”, being submitted by Sewan Das Patle for the award of the degree of **Doctor of Philosophy (Mechanical Engineering)** of NIT Rourkela, is a record of bonafide research work carried out by him under my supervision and guidance. Mr. Patle has worked for more than three years on the above problem at the Department of Mechanical Engineering, National Institute of Technology, Rourkela and this has reached the standard fulfilling the requirements and the regulation relating to the degree. The contents of this thesis, in full or part, have not been submitted to any other university or institution for the award of any degree or diploma.

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(Sewan Das Patle)

Date:

Abstract

Key Words:

(Thermophysical properties, Periodic heating, Temperature oscillation, Transient technique, Peltier heating, Liquids)

The problem of determining the thermophysical properties of material has been of great interest to scientists and engineers for more than hundred years. The thermophysical study of material has gained much importance recently with the vast development of newer materials. The development, specification, and quality control of materials used in semiconductor devices and thermal management often require the measurement of thermophysical properties where these data can be critical to a successful design.

Knowledge of thermal properties is essential in the efficient and economical design of all processing operations involving heat transfer. Problems of heat removal in processes involving electronics chips, laser applications, similar high-energy devices and the power generation industries, materials are selected primarily considering their thermal properties. Thermophysical properties are also of high interest for numerical modeling of heat transfer processes for better energy conversion and energy storage systems.

Due to the unique characteristics of liquid, measurement of thermal conductivity and thermal diffusivity are more challenging for liquids than for solids. Liquids do not maintain any fixed shape, and also can be easily changed compositionally, which alters their properties. The liquid cannot sustain a shear stress and also convection can occur in the presence of temperature gradients. These are the major error sources for many conventional techniques that measure liquid thermal conductivity and thermal diffusivity.

The well-known and conventional steady state method is the guarded hot plate technique. Now a days the transient techniques have achieved popularity over the conventional steady state methods because the transient technique

requires much less precise alignment, dimensional knowledge, short measuring time and stability. In addition to these, some of the transient methods bear less experimental errors due to its periodic nature. Some of the major advantages of the transient methods are that it gives a full set of thermophysical parameters in a single measurement, namely, specific heat, thermal diffusivity and thermal conductivity. Some of the well-known transient methods are hot wire method, transient hot strip method, transient plane source method, the laser flash method, thermal comparator method, temperature modulated differential scanning calorimeter (TMDSC), 3ω method, photo-thermal technique and temperature oscillation method.

In this work, a novel transient technique known as temperature oscillation method is presented. The principle of this technique was first proposed by Angstrom's in the year 1861 to measure thermal diffusivity and/or thermal conductivity of different metal strips with the application of steam water and cold water as a periodic heat source. The basic principle of this method is that if one end of the long bar shaped sample is heated periodically and other end is free to convect to an ambient temperature, then the temperature of the sample at a point also varies with the same period. The amplitude of the oscillation decreases exponentially along the length of the rod with a phase shift. The measure of these two properties of wave propagation can give the estimation of thermophysical properties of the sample.

This technique combines the advantages of a steady state measurement with the potential to measure a property describing a non-steady state. The method is purely thermal and the electrical components of the apparatus are away from the test sample, which does not influence the experimental data. One of the major advantages of the temperature oscillation technique is that a high degree of accuracy is attainable at different temperatures ranging from liquid helium to high temperatures upon which oscillation is imposed. Also the temperature oscillation method is related to its periodic character. During every period of such a process a whole cycle of the change is repeated. This makes it possible to considerably reduce random errors and to increase the signal to noise ratio. With the proliferation of modern computer based data acquisition systems, it is possible to design a

strategy for accurately computing thermophysical properties from the measured temperature data.

It is found from the literature that different investigators have derived different solutions depending on the boundary conditions, which are required for their experimental validation. A unified approach has been considered to derive a general governing equation, which accommodates a wide variety of boundary conditions. Under limiting cases this solution can be reduced to a particular boundary condition. The temperature oscillation is a pseudo-steady state process. Thus the experimental data are repeated after every cycle if the transients are absent. Thus the time elapsed to retrieve this steady state data is an important factor known as settling time. It can be demonstrated that the settling time for low thermal diffusivity and low heat transfer materials may be of the order of hours. Therefore, analysis of settling time has been carried to allow time lapse before the collection of steady state data in an experiment.

Uncertainty analysis is an essential component in an experimental procedure. The theory of uncertainty analysis has been presented. A step-by-step procedure to estimate the total uncertainty has been prescribed by taking the example of experimental data for a particular sample.

In the earlier method, the temperature oscillations are established by oscillating heat flux by sinusoidal resistance heating or by using steam water and cold water as a periodic heat source. A disadvantage of these types of heat sources is that the time average of the heat supplied during each cycle cannot be equal to zero. This leads to an increase in the mean temperature of the sample material. Thus Peltier element (or, Thermo Electric Cooler) has been adopted for the oscillating heat source. The excitation of Peltier element, whose one side is kept at a constant temperature, by a square wave leads to inaccurate prefixing of mean temperatures of oscillation. It has been demonstrated that a D.C. voltage modulated square wave excitation is preferred due to its better regulation of mean temperature of oscillation. The mean temperature of oscillation determines the temperature at which the thermophysical properties are to be estimated.

The amplitude attenuation is more sensitive to thermophysical property such as thermal diffusivity. Thus the amplitude attenuation is measured for the fundamental frequency of temperature oscillation by employing FFT technique. The

system considered for the measurement of thermophysical property is a semi-infinite model. The semi-infinite model in an experiment is ensured by inserting a thermocouple at the far end of the sample to indicate a non-oscillating signal output. Due to the unique characteristics of liquid, measurement of thermal properties is more challenging for liquids than for solids. Liquids cannot sustain a shear stress and convection can occur in the presence of temperature gradient are some of the major issues. Thus to demonstrate the measurement of thermal diffusivity by temperature oscillation method for a semi-infinite medium, four liquid samples, ethylene glycol, ethanol, glycerol and water have been selected.

The experimental results of the thermal diffusivity of the four liquid samples are obtained along with their uncertainties. The total uncertainty assessment has been used for plotting the error bar in the measurement of thermal diffusivity. The reliability of such is evident from its results as compared with the reported values. The deviation from the reported values in literatures are, 6.38%, 9.77%, 10.82%, and 2.74% for the respective samples of ethylene glycol, ethanol, glycerol and water. Due to the periodic nature of temperature oscillation method, the signal to noise ratio has been increased by reducing the random error. The minimum random error of 0.77% is observed for ethylene glycol whereas the maximum random error of 4.66% is obtained for glycerol. Due to the less contribution of random error, the total uncertainty exhibits a low value. Thus the simplicity in experimental procedures based on a lucid theory of temperature oscillation establishes an inexpensive measurement technique, which leads to achieve reliable results.

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Nomenclature

English Symbols

A	Amplitude ratio of steady periodic temperature
A_z	Complex amplitude ratio of steady periodic temperature
a_0, a_n	Fourier coefficients
B	Fixed or bias error
B_r	Bias limit
b	Defined by Equations (3.96) and (3.98)
b_n	Fourier coefficients
C, E, F, G	Defined by Equations (3.54) and (3.61)
C_1, C_1, \dots, C_{10}	Constant of integration
C_n	Complex Fourier coefficients
C_p	Specific heat at constant pressure, J/(Kg-K)
ΔT	Change in temperature
e_k	Precision error
f	Defined variable, $f = \sinh((\sinh^{-1} 2\delta)/2)$
h	Surface heat transfer coefficient, W/(m ² -K)
$I(\theta)$	Temperature solution for a unit signal input
$I(\theta_s)$	Steady periodic response for unit signal input
$I(\theta_T)$	Transient response for unit signal input
Im	Imaginary part of a complex quantity
i	Imaginary quantity, $i = \sqrt{-1}$
k	Thermal conductivity, W/(m-K)
L	Length of the bar, m
ℓ	Wavelength, m

M	Elemental error sources
m	Mass, kg
m_1	Function, $\sqrt{(s/\alpha) + (2h/rk)}$
m_2	Function, $\sqrt{(s/\alpha)}$
N	Number of repeated measurements
n	Indices
Q	Heat transfer, W
q	Heat flux, W/m ²
Re	Real part of a complex quantity
Res	Residue
r	Radius of the bar, m
S_r	Random limit
S_x	Precision index
$S_{\bar{x}}$	Average precision index
s	Laplace transform variable
s_n	Laplace poles
T	Temperature, K
T_a	Ambient temperature, K
T_{m1}, T_{m2}	Mean temperature of oscillation, K
T_0, T_L	Amplitude of periodic signal
T_{in}	Temperature amplitude at input
T_{out}	Temperature amplitude at output
t	Time, s
t_d	Student factor from t-distribution
t_p	Periodic time, s
\bar{X}	Average value of X
X	Measured value
x	Spatial variable, m
U_r	Total uncertainty

Greek Symbols

α	Thermal diffusivity, m^2/s
β	Function, $\beta = \sqrt{2h/rk}$
β^*	Dimensionless number, $\beta^* = x\sqrt{\omega/2\alpha}$
β_1	Function, $\sqrt{(i\omega/\alpha) + (2h/rk)}$
β_2	Function, $\sqrt{i\omega/\alpha}$
γ	Dimensionless parameter, $x(\omega/2\alpha)$
δ	Dimensionless parameter, $\alpha h/\omega r k$
δ_k	Total error
δT_{in}	Bias error in T_{in}
δT_{out}	Bias error in T_{out}
ε	Dimensionless spatial variable, x/L
κ	Wave number
θ	Temperature difference
θ_{m1}	Temperature difference, $T - T_{m1}$
θ_{m2}	Temperature difference, $T - T_{m2}$
$(\theta_T)_R$	Amplitude of transient temperature
$\bar{\theta}$	Laplace transform of θ
ν	Degrees of freedom
ρ	Defined by Eq. (4.22)
$\bar{\rho}$	Density, kg/m^3
$\varphi_{x,t}$	Solution of transient problem
$\bar{\varphi}$	Laplace transform of $\varphi_{x,t}$
Ψ_x	Steady state solution
Φ	Phase change, radian
Φ_0, Φ_L	Phase, radian
ω	Angular velocity, radian/s

ω^*	Dimensionless number, ωt
ζ	Dimensionless number, α/tL^2
ζ_s	Dimensionless settling time
τ	Relaxation time

Subscripts

0, m_1	At $x = 0$
L, m_2	At $x = L$
s	Precision limit
S	Steady state part
T	Transient part

Abbreviations

CF	Complementary Function
FFT	Fast Fourier Transform
PI	Particular Integral
TEC	Thermo Electric Cooler
TMDSC	Temperature Modulated Differential Scanning Calorimeter
TOM	Temperature Oscillation Method

Chapter 1

Introduction

1.1 General

The problem of determining the thermophysical properties of material has been of great interest to scientists and engineers for more than hundred years. The thermophysical study of material has gained much importance recently with the vast development of newer materials, rapid industrial development, and growth of material research and advent of new measuring technologies. Thermophysical properties are also of high interest for numerical modeling of heat transfer processes, for better energy conversion and energy storage systems. This data can be critical to a successful design; especially with the rapidly increasing cooling requirements that result from the packaging of higher performance devices.

Knowledge of thermal properties is essential in the efficient and economical design of all process operations involving heat transfer. Some of the common processes involving heat transfer are heating, cooling, freezing, cutting and drilling, annealing, cladding, etc that may set up thermal stresses in the material or alters its microstructure. In energy conversion system such as atomic or nuclear reactor the thermal analysis is indispensable. However, for most pure materials and mixtures there is still a lack of data for thermophysical properties in the literature.

In order to achieve high performance and reliabilities in energy conversion and utilization, thermal analysis for controlling the transport of heat is essential. Therefore knowledge about thermophysical properties of material and the

mechanism of heat transfer inside these materials are indispensable. The accuracy of thermal engineering calculation depends on the accuracy to which the thermophysical properties are known. Numerous examples could be cited of flaws in equipment and process design or failure to meet performance specification that were attributable to misinformation associated with the selection of key property values used in the initial system analysis. Selection of reliable property data is an integral part of any careful engineering analysis.

Thermophysical properties, such as thermal conductivity and thermal diffusivity, are very important properties. Accurate values of these properties are critical for practical engineering design as well as theoretical studies and analysis, especially in the fields of heat transfer and thermal processing. As both the thermal conductivity and thermal diffusivity are properties that characterize the heat transfer behavior, applications including conduction and convection heat transfer, heat exchanger design, and insulation problems, etc., heavily rely on the availability of these two parameters.

Due to the unique characteristics of liquid, measurement of the thermal conductivity and thermal diffusivity are more challenging for liquids than for solids. Liquids do not maintain any fixed shape, and can be easily changed compositionally, which alters their properties. Also, since liquids cannot sustain a shear stress, convection can occur in the presence of temperature gradients in the liquid, which is one of the major error sources for many conventional techniques that measure liquid thermal conductivity and thermal diffusivity.

1.2 Role of Heat Transfer in Various Fields of Engineering

The subject of heat transfer is of fundamental importance in many branches of engineering. A mechanical engineer may be interested in knowing the mechanisms of heat transfer involved in the operation of equipment, in a thermal power plant in order to improve their performance. Nuclear power plants require precise information on heat transfer, as safe operation is an important factor in their design. Refrigeration and air-conditioning systems also involve heat-exchanging devices, which need careful design. Electrical engineers are keen to avoid material damage due to hot spots, developed by improper heat transfer design, in electric motors, generators, transformers, and in other electrical appliances. As the

miniaturization of integrated circuits are advancing in a rapid rate, the electronics and computer engineers are interested in heat dissipation from semiconductor devices and circuit boards so that these devices can operate within its specified temperature limit. Chemical engineers are interested in heat transfer processes in various chemical reactions. In metallurgical field, engineers would be interested in knowing the rate of heat transfer required for a particular heat treatment process, for example, the rate of cooling in a casting process has a profound influence on the quality of the final product. Aeronautical engineers are interested in knowing the heat transfer rate in rocket nozzles and in heat shields used in re-entry vehicles. An agricultural engineer would be interested in the drying of food grains, food processing and preservation. A civil engineer would need to be aware of the thermal stresses developed in quick-setting concrete, the effect of heat and mass transfer on building and building materials and also the effect of heat on nuclear containment, etc. An environmental engineer is concerned with the effect of heat on the dispersion of pollutants in air, diffusion of pollutants in soils, thermal pollution in lakes and seas and their impact on life.

The study of heat transfer provides economical and efficient solutions for critical problems encountered in many engineering items of equipment. For example, one can consider the development of heat pipes that can transport heat at a much greater rate than copper or silver rods of the same dimensions, even at almost isothermal conditions. The development of present day gas turbine blades, in which the gas temperature exceeds the melting point of the material of the blade, is possible by providing efficient cooling systems and is another example of the success of heat transfer design methods.

Although there are many successful heat transfer designs, further developments are still necessary in order to increase the life span and efficiency of many devices discussed previously, which can lead to many more new inventions. Thus the studies of thermophysical parameters, which directly control the heat transfer processes, are of interest in almost all the fields of engineering.

1.3 Thermal Properties and its Role in Heat Transfer

The needs of thermal properties to describe material that are generally considered in the context of heat transfer are: thermal conductivity, thermal

diffusivity, specific heats, and surface heat transfer coefficient etc. Heat transfer coefficient is not a thermal property of a material but it is influenced by thermal properties.

Thermal conductivity:

The material property governing the flow of heat through a material at steady state is the thermal conductivity, k (W/mK); thermal conductivity is a measure of the ability of a material to conduct heat. In general, the thermal conductivity is primarily dependent on composition, but also on the other factor that affects the heat flow paths such as porosity and shape, size and arrangement of void spaces, homogeneity, fibers and their orientation. The highest conductivities are those of diamond, silver, copper and aluminum. The lowest are shown by highly porous materials like firebrick, cork, and foams, in which conductivity is limited partly by the gas entrapped in the porosity.

Specific heat:

The specific heat of a material is defined as the amount of energy required to raise one unit of temperature of one unit mass of material at constant pressure.

$$C_p = \frac{Q}{m \Delta T} \quad (1.1)$$

Thermal diffusivity:

The property governing transient heat flow is the thermal diffusivity, α (m^2/s). Transient heat transfer problems occur when the temperature distribution changes with time. The fundamental quantity that enters into heat transfer situations not at steady state is the thermal diffusivity. Thermal diffusivity relates the ability of a material to conduct heat to store. It is related to the steady state thermal conductivity through the equation

$$\alpha = \frac{k}{\bar{\rho} C_p} \quad (1.2)$$

Where α is the thermal diffusivity, k is the thermal conductivity, C_p is the specific heat, and $\bar{\rho}$ is the density. The diffusivity is a measure of how quickly a body can change its temperature, it increases with the ability of a body to conduct

heat k and it decreases with the amount of heat needed to change the temperature of a body C_p ; all three quantities on the right hand side of Eq. (1.2), as well as the thermal diffusivity, can be functions of temperature.

1.4 Heat Conduction Models and its Boundary Conditions

The basic thermal properties, which govern the heat transfer by conduction, are thermal conductivity, thermal diffusivity and specific heats. The heat transfer coefficient and relaxation time are not thermal properties of a material but they are influenced by the heat conduction process and the associated boundary conditions. The role of relaxation time suggests that there is a time lag between heat flow and temperature gradient. The heat flow does not start instantaneously but grows gradually with the relaxation time after the application of a temperature gradient. The relaxation time classifies the heat conduction to hyperbolic and parabolic models. The parabolic model predicts that the temperature disturbances propagate at an infinite speed where as in hyperbolic model the propagation speed is finite.

1.4.1 Parabolic Model

Parabolic heat conduction analysis is based on two basic relations, namely, the first law of thermodynamics and the Fourier law. In classical heat conduction theory, the constitutive governing equation for heat flow is given by Fourier's law,

$$q = -k \frac{\partial T}{\partial x} \quad (1.3)$$

This states that the heat flux q is proportional to the temperature gradient, where k is the thermal conductivity. The first law of thermodynamics for the heat flow can be stated as,

$$\rho c_p \frac{\partial T}{\partial t} = -\frac{\partial q}{\partial x} \quad (1.4)$$

When the constitutive relation, as expressed by Eq. (1.3) is incorporated into first law of thermodynamics, the resultant equation yields the parabolic partial differential equation for heat conduction as,

$$\frac{\partial T}{\partial t} = \alpha \frac{\partial^2 T}{\partial x^2} \quad (1.5)$$

It may be observed that in the constitutive Eq. (1.3), there is no involvement of time parameter. The heat flows instantaneously with the application of temperature gradient. This results an infinite heat propagation velocity, which is unrealistic in many situations. This invokes the hyperbolic heat conduction model. However, at normal conditions the parabolic model is adequate since the velocity of thermal wave is very large.

1.4.2 Hyperbolic Model

Fourier heat flux predicts that heat conduction is a diffusion phenomenon in which temperature disturbances will propagate at infinite velocities. This is physically unreasonable. In order to eliminate this anomaly of infinite heat propagation velocity and to account for the presence of the observed thermal waves a more precise heat flux law needs to be postulated. Since it is desired to retain the basic notion of first law of thermodynamics as described by the equation (1.4). Vernotte [20] suggested a modified heat flux law in the form

$$\tau \frac{\partial q}{\partial t} + q = -k \frac{\partial T}{\partial x} \quad (1.6)$$

Although the constitutive heat flux equation are different for the hyperbolic and parabolic models, the energy equation remains unchanged and is given by Eq. (1.4). The hyperbolic model is characterized by the addition of relaxation time, τ . When a heat flux of the form given by Eq. (1.6) is used in conjunction with first law of thermodynamics, Eq. (1.4), a hyperbolic heat conduction equation results in the form

$$\tau \frac{\partial^2 T}{\partial t^2} + \frac{\partial T}{\partial t} = -\alpha \frac{\partial^2 T}{\partial x^2} \quad (1.7)$$

For $\tau \rightarrow 0$, the parabolic and hyperbolic models coincide with each other. In hyperbolic model τ represents relaxation or start up time for the commencement of heat flow after a temperature gradient has been imposed. It means that the heat flow does not start instantaneously but grows gradually with a relaxation time, after applying a temperature gradient. This hyperbolic equation becomes the governing equation for heat conduction when the propagation velocity of thermal waves is finite.

Transient heat conduction equations such as parabolic and hyperbolic are found in the most literature. These two equations are differentiated on the basis of propagation of heat wave inside the medium. The difference between the hyperbolic and parabolic heat conduction can be simply stated as follows: parabolic heat conduction involves an instantaneous increase in surface temperature and an infinite rate of energy diffusion, while hyperbolic heat conduction involves a gradual increase in surface temperature and finite rate of energy diffusion. Thus, the classical parabolic heat conduction equation successfully predicts the temperature and heat flux distribution for most practical purposes, except for extremely short times and for temperature near absolute zero.

In conventional material processing, the parabolic heat conduction equation is adequate to model heat transfer in the material because the temperature gradient involved in such a processes is moderate. Under steady state conditions, the hyperbolic model reduces to the Fourier model even when relaxation time is not zero; hence the temperature predicted by the two models (parabolic and hyperbolic) will differ only under non-steady state conditions. When surface radiation is involved the hyperbolic solution, as a result of the higher surface temperatures, loses more energy due to radiation than the parabolic solution. Thus with increased surface radiation, the time to converge the difference between the hyperbolic and parabolic temperature profiles decreases. In general, the non-Fourier effect is shown to decay quickly. It has been observed that for most practical conduction problem the finite speed of propagation in hyperbolic equation is very large compared to the diffusivity.

- **Boundary Conditions**

The differential equations governing heat conduction will be complete for any problem only if the appropriate initial and boundary conditions are specified. With the necessary conditions, the solution to heat conduction equation is unique and feasible. It should be observed that the heat conduction equation has second-order terms and hence requires two boundary conditions. Since time appears as a first-order terms, only one initial value (i.e., at some instant of time all temperatures must be known) needs to be specified for the entire body. This instant of time may be taken as the origin of time coordinate and thus termed out as initial condition. It

may be further stated as initial condition specifies the distribution of temperature at the origin of the time coordinate, whereas boundary conditions specify the thermal condition at the boundary surfaces of the region.

The various common types of boundary conditions that may arise in heat conduction problems may be classified into three kinds. If the temperature at the surface (or the boundary) is given, the resulting boundary condition is called the Dirichlet condition or the boundary condition of first kind. It can be expressed mathematically as,

$$T = T_0 \text{ on } \Gamma_T \quad (1.8)$$

In many physical situations, it is possible that the heat flux rather than the temperature is specified at the boundary. The resulting boundary condition is mathematically expressed as,

$$q = -k \frac{\partial T}{\partial n} = C \text{ on } \Gamma_{qf} \quad (1.9)$$

The boundary condition of the above type, given by Eq. (1.9) is called the Neumann condition or boundary conditions of the second kind.

Another type of situation may arise when the body loses (or gains) heat by convection while it is in contact with a fluid at a given temperature T_a . The convective heat transfer coefficient h is also given. The mathematical form of this type of boundary condition is given by

$$-k \frac{\partial T}{\partial n} = h(T - T_a) \text{ on } \Gamma_{qc} \quad (1.10)$$

The form of the boundary condition given by Eq. (1.10) is called the Rabin condition or a boundary condition of the third kind or a convection boundary condition.

In Equations (1.8) to (1.10), T_0 is the prescribed temperature; Γ the boundary surface; n is the outward direction of normal to the surface and C is the given constant flux. The insulated or adiabatic, condition can be obtained by substituting $C = 0$. The suffices T , qf and qc denote temperature, heat flux and heat convection respectively.

Another thermal boundary condition that frequently arises in nature as well as in engineering systems is the periodic change of the temperature of the

surroundings. That is: (1) the daily and seasonal changes of solar radiation on the soil or on buildings, (2) the periodic changes in the cylinders of internal combustion engines, (3) various experimental temperature controls by a thermostat, and the periodic heat transfer in a regenerator. For theoretical and experimental modeling of the transient techniques, wide variation of sample size and boundary conditions can be classified as:

- (i) Long sample with one end of the sample is sinusoidal.
- (ii) Short sample (finite length) with the boundary conditions at the two ends are:
 - a. Sinusoidal - adiabatic
 - b. Sinusoidal - isothermal
 - c. Sinusoidal - sinusoidal

Many methods have been developed to analytically solve the heat conduction equation with known periodic oscillation boundary condition accurately. Those are pseudo-steady state method, variable separation method, splitting boundary condition method and direct Laplace transform technique. The temperature solution of the problems is usually composed of both steady periodic and transient decaying parts. The transient part decays to zero in the limit of long time. Pseudo-steady state solution method gives only periodic steady state solution and all other techniques give complete solutions, both steady periodic and transient. Pseudo-steady state technique is applicable only for the solution of homogeneous type of boundary condition. Non-homogeneous boundary value problem can be solved directly using Laplace transform technique or first splitting the boundary condition then applying either variable separation method or Laplace transform technique. A complete solution of transient heat conduction equation is obtained by the methods namely variable separation method; splitting boundary condition method and direct Laplace transform method. The amplitude ratio and phase change between outputs to input is obtained from steady periodic solution. Evaluation of these two properties gives measurement of thermophysical parameter.

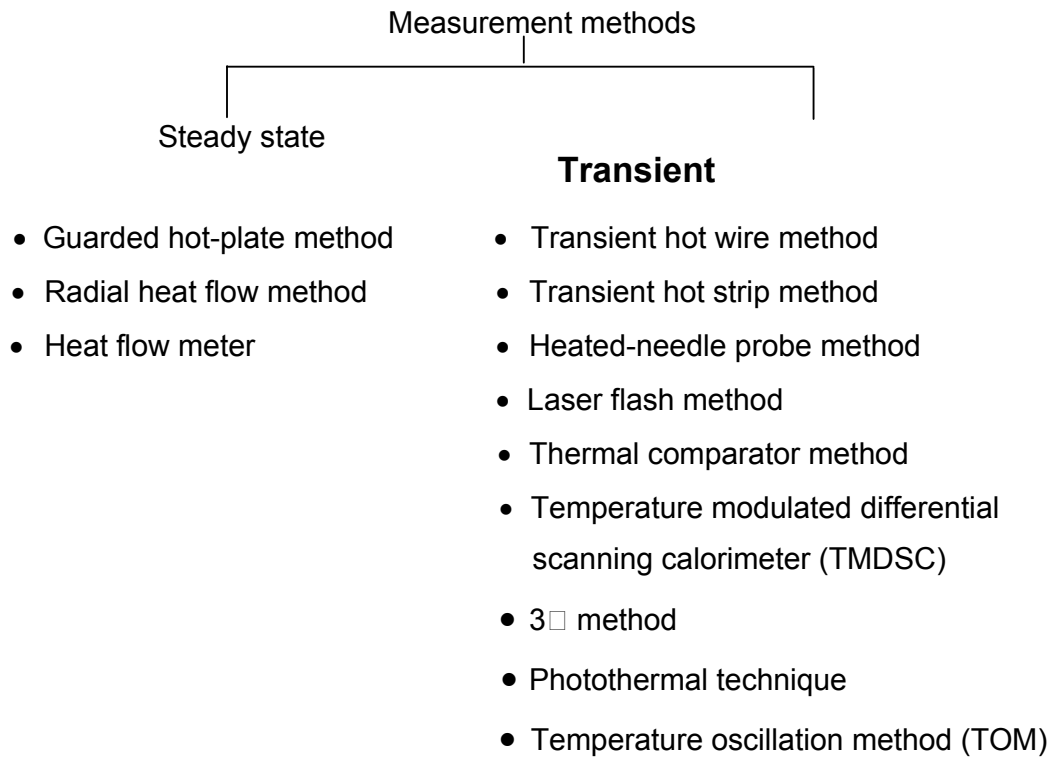
Although a number of analytical solutions for conduction heat transfer problems are available (Carslaw and Jaeger [1], Ozisik [2]) in many practical situations, the geometry and the boundary conditions are so complex that an

analytical solution is not possible. Even if one could develop analytical relations for such complicated cases, these will invariably involve complex series solutions and would thus be practically difficult to implement. In such situations, conduction heat transfer problems do need a numerical solution. The commonly employed numerical methods are the Finite Difference and Finite Volume methods.

1.5 Measurement Techniques of Thermal Properties

Over the years, a number of methods have been developed to measure thermal transport properties of many different materials. To keep up with the quick development of new materials and the ever-increasing importance of accommodating new applications, better accuracy and precision of measurement, variations of older methods and the introduction of completely new techniques have been more common recently. Now a days, it is often not enough to get approximate data from textbooks, but measurements of real samples are necessary. Small variation in composition, processing parameters and utilization conditions change the behavior and properties, and if new materials are to be used at their optimum potential, accurate measurements are essential. The use of short measuring time is perhaps the most distinctive feature of the any experimental method. Therefore, in designing an apparatus, accurate and rapidly responding instruments are essential. Advantages in instrumentation can thus help to increase the versatility and overall accuracy of the method so that a wide range of materials can be studied.

In general, the measurement techniques of thermophysical properties are mainly classified into steady state (stationary) and non-steady (transient) state methods. These methods can be further subclassified on the basis of basic principle of measurement; number of thermophysical properties measured simultaneously, suitability to test different materials, selection of heat source, output measuring device and various experimental conditions. Both categories of techniques provide a temperature gradient and then monitor the response of the material to this gradient. The various popular methods for measuring thermal diffusivity, specific heat, thermal conductivity, relaxation time, and surface heat transfer coefficient of different materials are classified as:



1.5.1 Steady State Method:

The essential constitutive equation for thermal conduction relates the heat flux in a material to the temperature gradient by the equation:

$$Q = -k\nabla T \quad (1.11)$$

It is not possible to measure local heat fluxes and gradients; thus all experimental techniques must make use of an integrated form of the equation, subject to certain conditions at the boundaries of the sample. All experiments are designed so that the mathematical problem of the ideal model is reduced to an integral of the one-dimensional version of Eq. (1.11), which yields, in general:

$$Q_a = Hk\Delta T \quad (1.12)$$

in which H is constant for a given apparatus and depends on the geometric arrangement of the boundaries of the test sample. Typical arrangements of the apparatus, which have been employed in conjunction with Eq. (1.12), are two flat, parallel plates on either side of a sample, concentric cylinders with the sample in the annulus and concentric spheres.

Techniques that make use of Eq. (1.12) are known as steady state techniques and they have found wide application. They are operated usually by measuring the temperature difference ΔT that is generated by the application of a

measured heat input Q_a at one of the boundaries. The absolute determination of the thermal conductivity, k , of the sample contained between the boundaries then requires knowledge of the geometry of the cell contained in the quantity H . In practice, it is impossible to arrange an exactly one-dimensional heat flow in any finite sample so that great efforts have to be devoted to approach these circumstances and then there must always be corrections to Eq. (1.12) to account for departures from the ideal situation.

One of the steady state methods is parallel-plate instrument as shown in Fig. (1.1). The sample is contained in the gap between two plates (upper and lower) maintained a distance d apart by spacers. A small amount of heat, Q_a , is generated electrically in the upper plate and is transported through the sample to the lower plate. Around the upper plate, and very close to it, is placed a guard plate. This plate is, in many instruments, maintained automatically at the same temperature as the upper plate so as to reduce heat losses from the upper surfaces of the upper plate and to most nearly secure a one-dimensional heat flow at the edges of the sample.

1.5.2 Transient method:

If the application of heat to one region of the test sample is made in some kind of time-dependent fashion, then the temporal response of the temperature in any region of the sample can be used to determine the thermal conductivity of the fluid. In these transient techniques, the fundamental differential equation that is important for the conduction process is:

$$\bar{\rho} C_p \frac{\partial T}{\partial t} = \nabla \cdot (k \nabla T) \quad (1.13)$$

where $\bar{\rho}$ is the density of the material and C_p its isobaric heat capacity. In most, but not all circumstances, it is acceptable to ignore the temperature dependence of the thermal conductivity in this equation and to write:

$$\frac{\partial T}{\partial t} = \frac{k}{\bar{\rho} C_p} \nabla^2 T = \alpha \nabla^2 T \quad (1.14)$$

in which α known as the thermal diffusivity.

Experimental techniques for the measurement of the thermal conductivity based on Eq. (1.14) generally take the form of the application of heat at one surface of the sample in a known time-dependent manner, followed by detection of the temperature change in the material at the same or a different location. In most applications, every effort is made to ensure that the heat conduction is unidirectional so that the integration of Eq. (1.14) is straightforward. This is never accomplished in practice, so some corrections to the integrated form of Eq. (1.14) are necessary. The techniques differ among each other by virtue of the method of generating the heat, of measuring the transient temperature rise, and of the geometric configuration. Interestingly, in one geometric configuration only, it is possible to determine the thermal conductivity essentially independently of knowledge of $\bar{\rho}$ and C_p , which has evident advantages. More usually, it is the thermal diffusivity α that is the quantity measured directly, so that the evaluation of the thermal conductivity requires further, independent measurements.

Some of the transient methods and technique (TOM) used in the present thesis are focused here. In transient hot wire technique, the thermal conductivity of a material is determined by observing the temporal evolution of the temperature of a very thin metallic wire (see Fig. 1.2) after a step change in voltage has been applied to it. The voltage applied results in the creation of a line source of nearly constant heat flux in the fluid. As the wire is surrounded by the sample material, this produces a temperature field in the material that increases with time. The wire itself acts as the temperature sensor and, from its resistance change; its temperature change is evaluated and this is related to the thermal conductivity of the surrounding material. The transient hot-disk instrument suitable for solid material is shown in Fig. (1.3). The sensor in this case comprises a thin metal strip, often of nickel, wound in the form of a double spiral in a plane. It is printed on, and embedded within, a thin sandwich formed by two layers of a material that is a poor electrical conductor but a good thermal conductor. This disk heater is then, in turn, placed either between two halves of a disk-shaped sample of solid or affixed to the outside of the sample. Heated-needle probe is shown schematically in Fig. (1.4) where it is seen that it consists of a thin, hollow, metallic needle (diameter 3 mm) containing an electric heater and a separate thermistors as a probe to record the

temperature history of the needle following initiation of a heat pulse. The temperature history of the probe is generally interpreted with the aid of the equation appropriate to a transient hot-wire instrument but in a relative manner whereby its response is calibrated against known standards. Fig. (1.5) contains a schematic diagram of the laser-flash instrument, as it is available today in the commercial form. The sample is illuminated on one face with of the sample causes the generation of heat at that front surface, which is subsequently transmitted throughout the sample to the back face of the sample where the temperature rise is detected with an infrared remote sensor. The interpretation of measurements is based on a one-dimensional solution of Eq. (1.14) subject to an initial condition of an instantaneous heat pulse at one location.

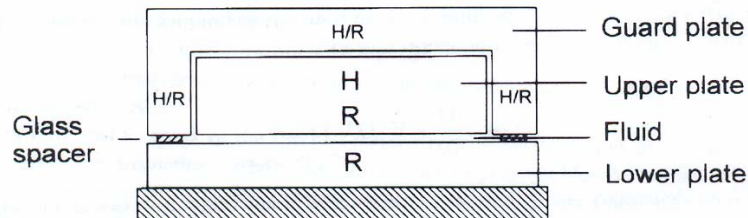


Figure 1.1 Schematic diagram of guarded parallel-plate instrument. (H = heater, R = resistance thermometer).

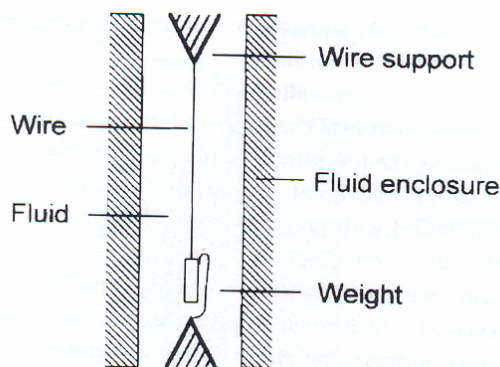


Figure 1.2 Schematic diagram of a transient hot-wire instrument for fluid.

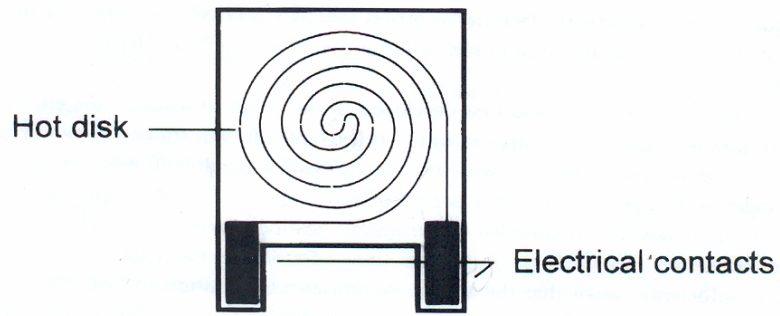


Figure 1.3 Schematic diagram of a transient hot-disk instrument.

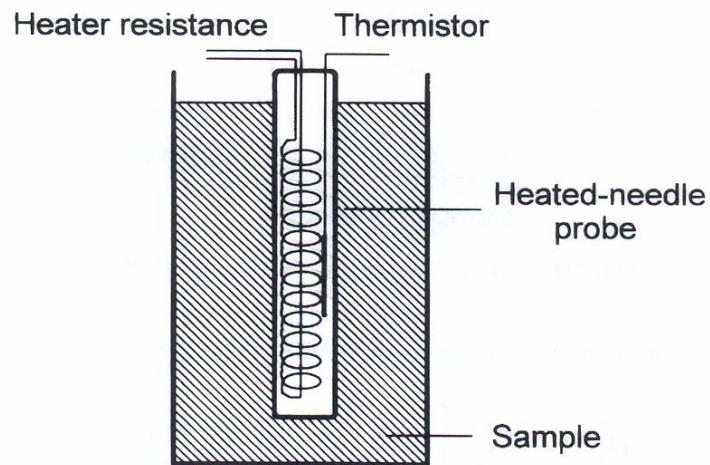


Figure 1.4 Schematic diagram of a transient heated-needle probe.

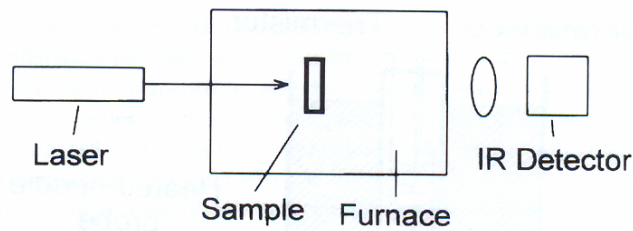


Figure 1.5 Schematic diagram of a laser-flash instrument.

1.5.3 Comparison Between Steady State and Transient Methods

The simplicity of stationary heat conduction processes, steady state method yields the capability of measuring thermal conductivity alone due to its inherent nature of involving a single thermophysical parameter in the basic governing

equation. The steady state method cannot provide the measurement of thermal diffusivity explicitly. Thus the other properties such as density and specific heat are to be found independently from another measurement to calculate the value of thermal diffusivity. Naturally, error propagation from the two measurements may lead to lower accuracy in the final result. Also measurement of thermal conductivity at higher and lower temperatures, the elimination of radiation losses is a major problem. However making the measurement in a time short enough minimizes this heat loss. It is a consolidated practice to estimate thermal diffusivity by transient method and indirectly estimate the thermal conductivity with specific heat and the density known a priori. This approach is often suggested because the thermal diffusivity measurement is usually less time consuming and more productive, if compared with the stationary techniques used for thermal conductivity measurements. These techniques for thermal conductivity evaluation require the heat flux measurement that is long, difficult to control and not much accurate.

The steady state technique poses two major problems for experiments with steam and water as heat sources. Firstly, the time required for the temperature distribution to reach a steady state value takes a long time. Secondly, moisture diffusion can occur from high temperature regions to low temperature regions, altering the composition of the sample. The moisture migration might be quite substantial because of the long test time. However, transient techniques eliminate both these problems as the test period mostly varies from a few seconds to a few minutes.

In comparison with steady state methods the advantage of the transient methods is that some of them give a full set of thermophysical parameters in a single measurement, namely, specific heat, thermal diffusivity, thermal conductivity. Measurement regime, data evaluation as well as specimen geometry has to be optimized for these transient methods to obtain stable results.

Moreover, the transient techniques, developed for thermal diffusivity measurement, require smaller sample dimensions and can operate in a wide temperature range. In fact, thermal diffusivity methods have been successfully applied from very low temperatures up to 3000⁰C. Another advantage of measuring the transient thermal diffusivity instead of the stationary thermal conductivity is related to the possibility of satisfying the condition of constant temperature during

the measurement, being typical temperature variations during the measurement smaller than 1-2°C.

One of the methods of transient technique is the temperature oscillation method. The basic principle of this method is the application of periodic heat source at the boundary. This results the temperature oscillation at a location in sample along its length with the same frequency of applied heat source. The measurement of amplitude and phase change of the temperature wave propagation can give the estimation of thermophysical properties. Now a days the transient technique based on temperature oscillation has achieved popularity for yielding less experimental error due to its periodic nature, simplicity, stability and accuracy of thermophysical property over a constant temperature.

1.5.4 Uncertainty Analysis

Since no measurement is perfectly accurate, means for describing inaccuracies are needed. An uncertainty is not the same as an error. An error in measurement is the difference between the true value and the recorded value; and error is the fixed number and cannot be a statistical variable. An uncertainty is a possible value that the error might take on in a given measurement. A somewhat “traditional” definition of the uncertainty of measurement is the following: “an estimate characterizing the range of values within which the true value of a measurement lies”. Hence, a measurement result without an accompanying statement of uncertainty is incomplete. It has little worth if no information is available on how correctly it describes that result. The advantages of uncertainty analysis of a proposed experiment can pay big dividends in the planning stage of an experiment, providing guidance for both the overall plan and for the execution of the details.

The total uncertainty in an observable or measurable quantity is decomposed into two parts: random errors and systematic errors. A random error is defined as the error detected by repeating the measurement procedure under the same conditions, while a systematic error is that which cannot be detected through this method and is usually associated with bias in experimental data. Random errors are generally caused by the imprecision of the measuring instruments and fluctuations in environmental conditions. They can, in general, be bound within

desired limits by using precision instruments and by controlling environmental factors. But systematic errors are inherent in the experimental process and a high degree of subjective judgment is necessary to estimate these errors. Calibration of the experimental system can eliminate some of these errors. In general the bias would be given by the difference between the mean (average) value of the N readings and the true value of the sample, whereas the random (precision) errors would cause the frequency of occurrence of the readings to be distributed about the mean value.

The uncertainty has different components, which may be categorized according to the method used to evaluate them. Each component of the uncertainty, however evaluated, is represented by an estimated standard deviation called standard uncertainty. Either the confidence level or the value of the coverage factor chosen has to be specified in addition to the expanded uncertainty range.

1.6 Objectives of the Present Investigation

There are many methods for the measurement of thermophysical parameters. Depending on the nature of the sample the methods often differ. The present investigation focuses the measurement principle of thermophysical properties by temperature oscillation. The basic principle of this method is that a periodic temperature oscillation applied at one end of the sample propagates the other end with amplitude attenuation and phase change. The measurement of these two quantities gives the estimation of thermophysical properties of the material. Truly speaking periodic temperature oscillation is a pseudo-steady state oscillation where this technique combines the advantages of a steady state measurement with the potential to measure a property describing a non-steady state. Due to this nature of periodic characteristic, whole cycle of the change is repeated. This increases the signal to noise ratio by reducing the random error.

There are different derivations related to the temperature oscillation and its boundary conditions in the literatures. A unified approach to derive a general governing equation, which accommodates a wide variety of boundary conditions, is an important feature of this investigation. It has been demonstrated that solution for a particular boundary condition can be derived from the generalized solution. In the temperature oscillation technique, the time to achieve the pseudo-steady state is an

important factor known as settling time. It may be noted that the settling time for low thermal diffusivity and low heat transfer coefficient materials may be of the order of hours. Therefore, analysis of this aspect of temperature oscillation is important to collect steady state data from an experiment.

The uncertainty analysis is an essential aspect in an experimental procedure. In this investigation, derivation of a general governing equation, settling time and uncertainty analysis have been given prime importance in the context of temperature oscillation technique which are lacking in literature.

In the earlier methods, the temperature oscillations are established by oscillating heat flux by sinusoidal electrical heating or by using steam water and cold water as periodic heat sources is that the time average of the heat supplied during each cycle can not be equal to zero. Thus leads to increase in mean temperature of sample material. The use of thermo electric cooler for heat sources can alleviate the heat balance problem.

Thus in the experimental front, Peltier element (Thermo-Electric Cooler) has been adopted for the oscillating heat sources with certain modification. The earlier approach of exciting Peltier element by a pure square wave has been eliminated. In the pure square wave technique, the mean temperature, at which the thermophysical properties is to be determined, can not be prefixed but this mean temperature can be determined as a post factor. Also, in pure square wave generator the amplitudes of higher harmonics are dominant compared to the fundamental frequency. This inherent drawback has been eliminated by exciting Peltier element by an electrical signal of small amplitude square wave modulated over a D.C. voltage. In this technique, the mean temperature of oscillation can be accurately prefixed by regulating the D.C. voltage.

In this investigation, a semi-infinite medium is considered and the amplitude measurement has been done for the fundamental frequency by utilizing FFT technique. The amplitude attenuation gives the estimation of thermal diffusivity of a sample. To demonstrate the performance of temperature oscillation method by adopting the above cited points four liquid samples, ethylene glycol, ethanol, glycerol and water have been selected.

In brief, the present investigation based on temperature oscillation method aims to study the following factors.

- To derive a generalized governing equation, which accommodates a wide variety of boundary conditions found in literature.
- To deduce the specific solution from the generalized solution.
- To determine the settling time to collect the steady state data from the experiment. To carry out the uncertainty analysis for the estimation of experimental error.
- To estimate the thermal diffusivity of liquid samples experimentally and comparison of results with the published work for the performance evaluation.

1.7 Organization of the Thesis

- The thesis contains six chapters including the present chapter (Chapter1) on introduction. In this chapter, the significance and methodology applied to estimate the thermophysical properties are discussed.
- A detailed survey of relevant literature is presented in Chapter 2. This includes various measurement techniques and their relative merits and demerits.
- In Chapter 3, a generalized solution is derived, which accommodates a wide variety of boundary conditions found in literature. For this case, periodic temperature oscillation with the constant angular frequency, but with different amplitude and phases are considered. The derivation of particular solutions for different boundary conditions from this general solution has been illustrated. Expressions for different possible practical situation in finite sample are presented.
- Chapter 4 is devoted to the theoretical principles for the measurement of thermophysical properties, which has been experimented in Chapter 5. In this chapter mathematical model, settling time and error analysis have been presented to supplement the estimation of thermal diffusivity in the Chapter 5.
- Chapter 5 presents the details of the experimental setup, which is custom-designed and built for this study. The experimental data of periodic temperature oscillation response for four liquid samples: ethylene glycol,

ethanol, glycerol, and water are presented. The thermal diffusivities of the test liquids are calculated along with their uncertainty analysis. These experimental results are compared with the other reported result in literature.

- Chapter 6 reports the general conclusions on the results and adopted methodology. The scope for future work has also been highlighted.

Chapter 2

Literature Review

2.1 Introduction

This chapter presents a review of the literature. It can be broadly classified under three categories. The first part of the survey deals with the theoretical solutions of the governing equations under different periodic boundary conditions. The various analytical techniques adopted in the literature are variable separation method, Green function solution and Laplace transform technique. The second part of the survey deals with some common methods employed for measurement of thermal conductivity and/or diffusivity of materials. These methods can roughly be divided into two methods, namely, steady state and transient. Various popular methods are guarded hot plate method, hot wire method, laser flash method, thermal comparator method, temperature-modulated differential scanning calorimeter (TMDSC), 3ω method, photo-thermal technique and temperature oscillation method. The third part of the survey deals with the uncertainty evaluation technique associated in the experimental result. The investigation reported in this thesis uses the temperature oscillation method. The literature pertaining to this method for measurement of thermophysical property is treated elaborately.

2.2 Analytical Solutions

The solution of transient heat conduction equation with associated boundary conditions has been the main goal of many analytical investigators. In

literature, hyperbolic and parabolic models have been used as governing equations by different investigators. A considerable literature on these subjects has grown up over the past hundred years. Some of the relevant literature has been presented in this section.

2.2.1 Parabolic Model Solution

Parabolic heat conduction involves an instantaneous increase in surface temperature and an infinite rate of energy diffusion. The classical parabolic heat conduction equation successfully predicts the temperature and heat flux distribution for most practical purposes, except for extremely short time and for temperature near absolute zero. Also, under steady state conditions, the hyperbolic model reduces to the Fourier or parabolic model even when relaxation time is not zero; hence the temperature predicted by the two models (parabolic and hyperbolic) will differ only under non-steady state conditions. In general, the non-Fourier or hyperbolic effect is shown to decay quickly and the conventional Fourier equation is accurate after a short time of the initial transient.

A number of analytical solutions of conventional transient conduction equations with its prescribed boundary conditions (i.e. constant temperature, heat flux) are available in text of heat conduction [1,2]. The practical importance of heat conduction problems with periodic boundary condition was discussed by Carslaw and Jaeger [1], who cited applications in geophysics, experimental measurement of thermal diffusivity, and thermal stresses in cylinder walls of internal combustion engines. However, studies of periodic boundary conditions are few. Earlier theoretical studies on periodic boundary conditions were limited to one-dimensional semi-infinite bar. However, the sinusoidal wave generation in the materials has further motivated engineers and scientists to explore some additional solutions of long and short bars. In that sense, Ables et al. [3] derived a solution for a finite rod when one end of the bar is exposed to sinusoidal temperature oscillation and other end is free to radiate into the black body environment. Tomokiyo et al. [4] estimates thermal diffusivity of solids when one end is changed sinusoidally with constant angular frequency and the other end of the rod is connected to a heat sink maintained constant at a lower temperature. Lopez et al. [5] measured thermal diffusivity of semiconductors when one end is heated sinusoidally and other is

thermally isolated. In that analysis, heat losses from the lateral surfaces are also considered during the formulation of governing equation but an approximate derivation has been made for the expression of amplitude ratio and phase shift. Caulk [6] assumed temperature variations confined within a thin layer and calculated temperature distributions. This assumption is valid for a periodic boundary condition of high frequency. Zubair and Chaudhry [7] used Duhamle's theorem to solve the problem in a semi-infinite solid with time dependent surface heat flux. Czarnetzki et al. [8] have continued the development of temperature oscillation theory for simultaneous measurement of thermal diffusivity and thermal conductivity of solids, liquids and gases. For theoretical modeling, two geometries are considered. In semi-infinite body, the front surface is subjected to temperature oscillation over the mean temperature. In finite body, on each side of the slab, periodic surface temperature oscillations are generated with the same constant angular frequency, amplitude and phase. Complex steady periodic solutions are determined by applying Laplace transform technique. Czarnetzki et al. [9] derived a temperature solution for a thin circular plate of infinite radius and a strip specimen with point or line heat source oscillation at the center. Hajji et al. [10] used Green's function solution method for solving one-dimensional transient equation when the front surface of the slab is changed with periodic heat flux over the mean oscillation of heat flux and also, a sensitivity analysis has been done to show the set of parameters that can be adjusted to provide accurate results.

Infinite hollow cylinder geometry is considered for the solution of transient conduction equation with oscillating heating [11]. Pascale et al. [12] described an experimental study of heat transfer in oscillating flow inside a cylindrical tube. The inverse heat conduction principle is applied for the characterization of local heat transfers at the fluid–solid interface. Deepak Ganapathy et al. [13] used finite difference method to compute the thermal conductivity of composites with cylindrical particles. Jeong et al. [14] examined experimentally the influence of pulsation frequency on the convective heat transfer from a heated block array and evaluated the resonant frequency for convective heat transfer enhancement. Analyses of heat conduction mechanisms in suspended microstructures and uncertainty in measured thermal conductivity data are presented by Wenjun et al. [15]. In this study, steady state joule heating and electrical resistance thermometry

were used to measure lateral thermal conductivity of the suspended silicon/metal structures.

Chen et al. [16] proposes an efficient technique which combines the function specification method, the whole domain estimation approach and the linear least-squares-error method to estimate the unknown outer-wall heat flux and the inlet temperature simultaneously for conjugate heat transfer within a hydrodynamically developed turbulent pipe flow. Zueco et al. [17] solved the two-dimensional inverse heat conduction problem of estimating the time-dependent (or temperature-dependent) heat source in an orthotropic medium whose thermal properties are temperature-dependent. The solution of one-dimensional transient heat conduction equation for generalized boundary conditions has been developed by the method of residues and Laplace transform technique [18]. In this model, a finite circular bar is subjected to temperature oscillation at both the ends in the presence of heat loss from its lateral surface.

2.2.2 Hyperbolic Model Solution

With the advent of science and technology involving very low temperature near absolute zero (cryogenic engineering) or extremely short transient durations (laser heating) or very high heat fluxes in nuclear engineering, some investigators found that the heat propagation velocity of such a problem becomes finite. To account for the finite propagation velocity of thermal waves, a modified Fourier law of heat conduction has been proposed by Cattaneo [19], Vernotte [20] and Chester [21]. Ulbrich [22] has derived an electrical equation, which has an analogous form to Vernotte's hypothesis for hyperbolic heat conduction. According to this modified law, a new physical parameter, relaxation time was introduced. The relaxation time represents the time lag between the temperature gradient and the resulting heat flux vector. The concept of hyperbolic heat conduction equation has been also developed with several different approaches [23-27]. Since then, many analytic and numerical solutions of such a problems have been reported for semi-infinite and finite bodies under different initial and boundary conditions. Maurer and Thompson [28] emphasized the importance of the wave effect in response to a high heat flux irradiation. Kazimi and Erdman [29] investigated the interface temperature for two

suddenly contacting media. Wiggert [30] studied the characteristic method in thermal wave propagation.

Semi-Infinite Bodies:

Baumeister and Hamill [31] studied the temperature wave in semi-infinite solid subjected to a suddenly applied temperature changed at the wall. Amos et al. [32] solved the problem of uniform temperature whose boundary is suddenly subjected to an increase in temperature with time. Thermal pulse wave propagation in semi-infinite, one-dimensional solids was predicted by B. Vick et al. [33]. In his treatment, a volumetric energy source was used. Glass et al. [34] gave numerical solutions for a semi-infinite medium with surface radiation. The effect of temperature dependent thermal conductivity on the propagation of thermal wave was studied for a semi-infinite region under different boundary condition with a pulsed energy source by Glass et al. [35,36]. This effect can be important even at a long time after the initial transient if the thermal disturbance is oscillatory with the period of oscillation of the same order of magnitude as the thermal relaxation time [37]. A semi-infinite slab is considered for the solution of non-Fourier heat conduction with surface heat flux [38].

Finite Bodies:

Solution of the hyperbolic heat conduction equation for finite body under different initial and boundary conditions are available in literature. Ozisik et al. [39] gave an analytical solution in a finite slab with insulated boundaries. In his treatment, a volumetric energy source was used. Frankel et al. [40] using flux formulation of hyperbolic heat conduction equation gave an analytical solution for a finite slab under boundary condition of rectangular heat pulse. Kolesnikov [41] solved the transient heat conduction equation for generalized boundary conditions. Gembarovic et al. [42] gave an analytical solution for a finite slab under boundary condition of instantaneous heat pulse. Kar et al. [43] solved the problem with constant thermal diffusivity, where thermal conductivity, heat capacity and density are temperature dependent. Separation of variables and Laplace transforms are used in finding the solutions. Hybrid technique based on the Laplace transform and control volume methods can successfully be applied to suppress numerical oscillations in the vicinity of sharp discontinuities [44, 45]. Tang and Araki [46]

analyze a plane slab where the front surface is exposed to heat flux, while the rear surface is insulated.

Numerous papers dealing with such and other applications have appeared in literature in the recent past [47-50]. With more emphasis on precision material processing and operation with the above applications it is likely that an increase in the application of hyperbolic proposition will take place. These and many other studies have been very comprehensively reviewed by Ozisik and Tzou [51].

Various studies [52-55] report the appearance of a wave behavior in heterogeneous materials, in dielectric materials with sub micron thickness, and other applications. Minkowycz et al. [56] studied a similar wave like phenomenon in porous media. The generalized diffusion equation for conductors, non-conductors, and semi-conductors with sub micron structures are developed [57,58].

Recent Development:

Quaresma et al. [59] presented new heat-conduction equations, named ballistic-diffusive equations, which is derived from the Boltzmann equation. It showed that the new equations are the better approximation than the Fourier law and the Cattaneo equation for heat conduction at the scales when the device characteristic length, such as film thickness, is comparable to the heat-carrier mean free path and/or the characteristic time, such as, laser-pulse width is comparable to the heat-carrier relaxation time. A numerical technique with hybrid application of the Laplace transforms and control volume methods has been developed [60,61]. Temperature-dependent phase-lags are incorporated in the dual-phase-lag model to fully describe the experimental data of femtosecond laser heating on gold films of various thicknesses in the sub-micron range by Tzou et al. [62]. Certain anomalies in the analysis of hyperbolic equation for accurate measurement of temperature distribution are presented [63,64]. The lagging model with temperature-dependent thermal properties enables a consistent description of all the available experimental data for ultra fast laser heating on gold films. Pakdemirli et al. [65] solved hyperbolic heat conduction equation with temperature dependent thermal properties by employing approximate symmetry theory. Su et al. [66] studied the difference between the solutions of the phase lagging equation and the damped wave equation with considering one dimensional heat conduction in a thin,

homogeneous, constant cross section of finite rod where the front surface is exposed to heat flux, while the rear surface is insulated. Weixue et al. [67] presents a hybrid method to calculate direct exchange areas for an infinitely long black-walled rectangular enclosure. The hybrid method combines the finite volume method with the midpoint integration scheme. Ching [68] formulated a set of non-linear equation for the process of the inverse estimation. Lewandowska et al. [69] presents an analytical solution of the hyperbolic heat conduction equation for the case of a thin slab symmetrically heated on both sides.

2.3 Measurement Techniques

The methods to measure the thermophysical properties of materials are divided into steady state and transient methods. In the steady state method, constant heat sources are applied at the boundary when the temperature and heat sources are invariant with time. The mathematical model is a steady state equation. On the other hand the heat sources and temperature are time dependent in transient method and transient governing equations are used. Some of the popular measurement techniques are described in this section.

2.4 Steady State Methods

Methods that employ steady state measurement of thermal conductivity apply Fourier's law of heat conduction. A one-dimensional flow is employed most frequently with other geometrical arrangements. Common for all steady state methods is that the operator tries to establish a temperature gradient over a known thickness of a sample, and controls the heat flow from one side to the other.

The standard well-known guarded hot plate method [70] is based on the steady-state longitudinal heat flow principle, which determines the thermal conductivity of the material by applying Fourier's law. This method is generally used for measuring the thermal conductivity of samples that can be formed into a slab, whereas, radial heat flow method is more commonly used with powdered or granular material. Since steady state conditions may take several hours to establish, use of steam and water as heat sources may cause moisture migration. A schematic diagram is shown in Fig. (1.1), Chapter 1.

2.5 Transient Techniques

2.5.1 Transient Hot-Wire Technique

In this method, a metal wire of defined diameter and length is contacted in both ends by electrical conductors, placed in contact with the sample material, determines how the temperature rises in the wire. The temperature-time response is used to calculate the sample conductivity. Simplicity of hardware and design makes it an ideal method for some of the liquid samples. But measuring solid samples is very difficult, the reason being the contact resistance.

The method was first described in 1888 by Schiermacher, but its first practical application was reported in 1949 by Van der Held and Van Drunnen [71], who used it to measure the thermal diffusivity of liquid. However, its application to other materials was somewhat slower until in the late 1960s. The next step in the development of transient method is the hot strip method. This is used in non-electrically conducting materials, and the sample surfaces have to be rather smooth to give good readings. The further development of the strip is the transient plane source (TPS) technique. It is also called the Gustafsson Probe. The schematic diagram of transient hot-disk and heated probe has been shown in Figs. (1.3) and (1.4) respectively.

The transient hot-strip technique has been used to measure simultaneously thermal conductivity and thermal diffusivity of solids and liquids [72] and thermal conductivity of electrically conducting materials [73], effective thermal conductivity of copper powders [74]. Yimin Xuan et al. [75] used this technique for heat transfer enhancement of nanofluids. Perkins et al. [76] measured thermal conductivity of saturated liquid toluene by use of anodized tantalum by transient hot wire (THW) method at high temperatures. Different investigators [77-80] also used this technique to measure thermophysical properties of the sample. Although the hot-wire method has been widely used in practice, it has some shortcomings that are very difficult to overcome. One major difficulty is the effects of natural convection, which occurs due to buoyancy effects as the wire is continuously heated. In addition to this, temperature variation of the wire is measured using a bridge circuit in which the wire acts as one bridge. Accuracy of temperature measurement is thus decreased by electrical noise and drift problems within the bridge circuit. The poor

precision in the determination of the heating time also contributes to measurement errors.

2.5.2 Laser Flash Method

Parker et al. [81], in 1961 of the US Navy Radiological Defense Laboratory, first introduced the flash method. The flash method measures the diffusion time across a thin film by probing the temperature rise at the back surface resulting from a heat pulse delivered to the front surface. Lasers are frequently used as the heating source, in which case the method is known as laser flash. The technique has the very distinct advantages that it does not require physical contact between the test sample and the heater or detector. For this reason, it is a particularly appropriate technique for use at high temperatures or in aggressive environments. The method has widespread application to a wide range of materials, including solid homogeneous isotropic materials, composites, polymers, glasses, metals, refracting materials, insulating solids, and coatings.

The original laser pulse method of measuring thermal diffusivity proposed by Parker et al. [81] assumes ideal boundary and initial conditions, infinitely short laser pulse, and uniform heating of the sample face. The original concept has been gradually improved to account for real experimental conditions. In that sense, Donaldson et al. [82] extended Parker's flash method to high temperatures utilizing a laser pulse with radial heat flow. As an alternative to previous method, there appears to be potential advantages in utilizing a radial rather than axial heat flow for measuring thermal diffusivity. The literatures [83-90] summarize present-day knowledge related to an application of the flash method. Lazard et al. [91] described the coupled conductive-radiative transient heat transfer in a slab and presented a complete methodology to estimate the intrinsic diffusivity of semi-transparent samples. Benjamin et al. [92] also investigated the problem related to the coupling of conduction with convective and radiative heat transfer and described the parameter estimation procedure.

2.5.3 Thermal Comparator Method

The method is based on the well-known observation that when two materials at different temperatures are brought into contact over a small area, heat transfer

takes place from the hotter to cooler body, which is a function of the thermal conductivity of the materials. As a result, an intermediate temperature is very quickly attained at the point of contact. The contact temperature depends on the thermal conductivity of the two materials. This technique is used to measure thermal conductivity of bulk materials. Mukherjee et al. [93] used this method to measure thermal conductivity of organic liquids and liquid mixtures. Recently, Cheruparambil et al. [94] used the modified thermal comparator method to analyze the heat conduction process from the tip of the comparator to a very thin CVD diamond films.

2.5.4 Temperature Modulated Differential Scanning Calorimeter (TMDSC)

In this technique, the temperature is changed linearly with superimposed sinusoidal temperature modulation, and the sample thermal response is observed in comparison with that of the thermally inert reference material. The sample thermal response in the form of temperature difference is separated into the response in phase with the temperature modulation, and it is postulated that the in-phase response corresponds to reversible process, while the other components is due to non-reversible process. The purported advantages of TMDSC introduce the ability to separate overlapping phenomena, as well as improved resolution and sensitivity. There are problems associated with the interpretation of TMDSC data, especially when nonlinear processes, such as melting, reaction, or structural recovery are involved. Marcus et al. [95] measured the thermal conductivity of insulating materials in the range from 0.1 to 1.5 W/m °C which generally covers, polymers, ceramics and glasses. Takeo [96] has presented the applicability and limitation of TMDSC. Sindee et al. [97] derived the relationship between the thermal conductivity and the apparent heat capacity as a function of sample thickness, frequency, and the heat transfer coefficient. Using this method, the thermal conductivity of automotive polymers is measured by Ismat et al. [98]. Yoon [99] described the details of various types of modern Calorimetry.

2.5.5 3ω Method

In this method, the specimen itself serves as a heater and at the same time a temperature sensor, if it is electrically conductive and with a temperature dependent electric resistance, or for electrically nonconductive specimen, a metal strip is artificially deposited on its surface to serve both as the heater and the sensor. Lu et al. [100] measured the specific heat and thermal conductivity of platinum wire specimens at cryogenic temperatures. Borca et al. [101] used this method, in conjunction with a multilayer two-dimensional heat conduction model, to measure thermal conductivities of three antimonide-based materials.

2.5.6 Photo-Thermal Technique

Most non-contact measurements are based on photo-thermal techniques. Different photothermal techniques are available in the literature as transient thermo reflectance [102-106] or forced Rayleigh scattering [107], photothermal deflection technique [108-110], transient grating [111,112], and thermal wave cavity [113-116]. Many approaches have been adopted, with the significant differences being related to the method of heating, and the method of temperature detection. This can be classified depending upon whether thermal waves were generated by electrical [117] or optical heating and/or upon whether the phase lag and the amplitude were measured optically [118] or thermo-electrically. Analysis performed in support of these measurements usually differs in the temporal form of periodic heating-electrically [119] or laser pulsed modulated heating [120,121]. Photoacoustic microscopy uses photothermally generated acoustic waves to investigate subsurface structures and photothermal wave microscopy images thermal wave propagation and scattering by subsurface defects using probe beam. The main advantage of this method is that it is a non-contacting and non-destructive optical method. A disadvantage is that the poor availability of desirable optical properties of the material.

2.5.7 Temperature Oscillation Method (TOM)

The basic principle of this method is that if one end of a long bar shaped sample is heated periodically and other end is free to lose heat to an ambient, then the temperature of the sample at a point also varies with the same period. The

amplitude of the oscillation decreases exponentially along the length of the rod with a phase shift. The measure of these two properties of wave propagation can give the estimation of thermophysical properties of the sample. Using this principle, extensive work has been done to obtain the thermal properties of the materials.

The principle of this technique was first proposed by Angstrom [122] in the year 1861 to measure thermal diffusivity and/or thermal conductivity of a long bar of small cross section. Angstrom pointed out that the method yields the diffusivity directly, independently of the thermal conditions. He applied the theory of the method to determine the diffusivity of the soil from observations of soil temperature. Kelvin's also used the annual temperature changes in the earth at various depths to estimate the conductivity of the soil.

Billington [123] used Angstrom's method for measuring the thermal diffusivity of poor conductors of heat by plotting graphs between log of amplitude ratio verses length and time lag verses length. Sidles and Danielson [124] using a modification of this method first proposed by Angstrom have shown that a high degree of accuracy in the thermal diffusivity is attainable at temperatures as high as 1270K. They described an apparatus for measuring thermal diffusivity of metal wires about 20 in. in length. Nii [125] measured thermal diffusivities of semiconductors at temperatures, which did not exceed 380 °C. Green and cowls [126] measured thermal diffusivity of semiconductors by Angstrom's method. The apparatus is capable to measure the property between room temperature and 180C only. Savvides and Murray [127] described an apparatus for measuring the thermal diffusivity at high temperatures to 2% accuracy. The apparatus is capable of measuring the thermal diffusivity of a wide range of materials, such as single-crystal silicon, which has a high diffusivity, and low-diffusivity materials, such as Ge-Si alloys. A specially designed low-frequency sine-wave generator was employed to produce a very stable sinusoidal temperature wave. Gallego et al. [128] measured thermal conductivity of ribbon shaped carbon fibers by two different techniques. The results obtained with angstrom's method appear to be in closer agreement with those estimated by other technique. Pesty et al. [129] has designed experimental setup to generate sinusoidal temperature oscillation in ultrahigh vacuum. Morikawa and Hashimoto [130] measured thermal diffusivity of Ultra Thin Film of Polyimide by this method. Sarit Das, et al. [131] used the same measuring technique of

Czarnetzki and Roetzel to measure thermal conductivity enhancement of nanofluids. Morikawa and Hashimoto [132] used high-speed micro-scale infrared camera to the observation of thermal resistance at the interface of the multi-layer polymer films by using a temperature wave. This method is applicable of not only to determine the value of thermal conductivity or diffusivity but also to observe the two dimensional distribution or anisotropy of the thermal property of the materials. Ohmyoung et al. [133] presented a technique (STWM), which can image the phase lag and amplitude of thermal waves with sub-micrometer resolution by scanning a temperature-sensing nanoscale tip across a sample surface. This method is used to measure the amplitude and phase lag distribution at different frequencies on a sample. Nandi Putra et al. [134] studied the natural convection of nanofluids.

2.6 Merits and Drawbacks of Transient Techniques

The thermal comparator method is a technique for measuring thermal conductivity of bulk solids. Transient hot wire method is an ideal method for some liquids. One major difficulty of the hot wire method is the effect of natural convection. Also the accuracy is limited by the measuring precision of the temperature rise in the wire, which is another well-known obstacle to this technique. One disadvantage of the flash method is that access to the back surface requires thin freestanding samples and requires very precise measurement of small angles or phase difference. However, other methods have been developed to probe the front surface. A major draw back to the 3 ω technique is the extensive requirement of sample preparation, in contrast to most of the techniques using a laser as the heating source.

Some of the transient methods require expensive and/or large measurement setups. For example, the laser heating method requires very precise measurement of small angles or phase difference. The heated bar method requires elaborate preparation of samples, i.e., the deposition of thermocouples surfaces with silver filled epoxy. In contrast to other transient method, the temperature oscillation technique is relatively simple, inexpensive and compact, which leads to reliable and accurate results. This technique combines the advantages of a steady state measurement with the potential to measure a property describing a non-steady state. The method is purely thermal and the electrical components of the apparatus

are away from the test sample, which does not influence the experimental data. One of the major advantages of the temperature oscillation technique is that a high degree of accuracy is attainable at different temperatures ranging from liquid helium to high temperatures upon which oscillation is imposed. Also the temperature oscillation method is related to its periodic character. During every period of such a process a whole cycle of the change is repeated. The measurements of power input used to heat the system are not required. Because of this, absolute measurements of the temperature are not required so that only relative changes in magnitude of a temperature as a function of time and position must be recorded. The thermocouples used in this experiment therefore do not need to be calibrated. This makes it possible to considerably reduce random errors and to increase the signal to noise ratio. With the proliferation of modern computer based data acquisition systems, it is possible to design a strategy for accurately computing thermophysical properties from the measured temperature data. Although this method was primarily developed for measurement of homogeneous isotropic long bar solid materials, the method has now been successfully applied for estimation of advanced and more complex materials. Literature summaries present day knowledge related to the applications of this method to semitransparent materials, an isotropic media, layered structures, composites and ultra thin films and nano fluids.

2.7 Uncertainty Analysis

The difference between the measured value of a property and its true value is called measurement error. Any measurement is affected by errors due to a multitude of factors; the imperfection of the instruments used and of the measurement methods followed, the influence of the external perturbation, the variation of the ambient conditions, the subjectivity of the operator, etc. An available result from an experiment has a little worth if no information is available on how correctly it describes that property. For this purpose, the measurement of uncertainty is the most widely accepted concept. Hence it has been felt to include a section on the available literature.

Kline and McClintok [135] have proposed a procedure for estimating the uncertainty of the measured quantity in experimental studies. Single sample

uncertainty analysis has been described in the literature by the work of Moffat [136]. Moffat [137] described the use of a priori uncertainty analysis in choosing proposed techniques with examples. The purpose of uncertainty analysis is outlined by Kline [138]. The techniques of multiple sample analysis are described by Abernathy [139]. Moffat [140] outlines a conceptual bias, which includes a residual uncertainty due to variability arising in the true definition of the measured variable. Coleman and Steele [141] have well documented the experimental uncertainties and discussed its importance in planning and evaluating experiments. Estimation of uncertainty in thermal systems analysis and design is briefly described in literature [142,143]. Hall et al. [144] has made a program for the evaluation of uncertainty by an automatic differentiation technique. Adeyinka et al. [145] evaluated experimental uncertainty of measured entropy production.

Chapter 3

Derivation of General Theory of Temperature Oscillation

3.1 Introduction

Many experimental methods have been developed over the past centuries to measure the fundamental thermal properties of materials. One important class among them is the transient techniques where temperature oscillation method is widely due to its experimental simplicity and less random error. In earlier studies different investigators have proposed different solutions based on the different boundary conditions to validate their results with experiments. Infact, the boundary conditions used in a experiment are used to solve the differential equation and the solution is mapped to estimate the thermophysical properties. In this chapter an attempt has been made to present a generalized solution which accommodates a wide variety of boundary conditions. In this analysis a finite circular bar with lateral heat transfer is considered subjected to oscillation of same frequency but different amplitude and phase at both the ends. For simplicity in derivation, the boundary conditions are considered to be complex exponential quantities where its real and imaginary parts yield cosine and sine wave respectively.

An exact expression to determine the thermophysical properties by measuring the real amplitude ratio and phase shift of the propagating thermal

waves along the specimen has been derived. In an adiabatic situation (no lateral surface heat loss) either amplitude ratio or phase change lead to the estimation of one parameters such as thermal diffusivity. In this analysis the general solution is reduced to simplified solution depending on the boundary conditions.

3.2 General Governing Differential Equation with Lateral Heat Transfer

The analytical method for solution of the temperature distribution in a finite circular bar is developed subjected to temperature oscillation at both the ends in the presence of heat loss from its lateral surface.

A finite circular bar of length L and radius r , initially at a uniform temperature of T_a is taken into consideration. The general differential equation and its boundary conditions are described by Eqs. (3.1) to (3.1c). One can obtained the solution of one-dimensional transient parabolic heat conduction equation for a finite rod, which is initially at a uniform temperature. For times $t > 0$ the boundary surfaces at $x = 0$ and $x = L$ are imposed with sinusoidal temperature oscillation with same constant angular frequency but different amplitude and phase shift.

Assuming constant thermophysical properties k and α and no internal heat generation the unsteady one-dimensional formulation for this problem is written as:

$$\frac{\partial^2 T}{\partial x^2} = \frac{1}{\alpha} \frac{\partial T}{\partial t} + \frac{2h}{rk} (T - T_a) \quad (3.1)$$

The initial and boundary conditions can be expressed as,

$$T = T_a \quad \text{at} \quad t = 0, \text{ for } 0 \leq x \leq L \quad (3.1a)$$

Boundary conditions

$$T = T_{m1} + T_0 \exp(i(\omega t + \Phi_0)) \quad : x = 0, t > 0 \quad (3.1b)$$

$$T = T_{m2} + T_L \exp(i(\omega t + \Phi_L)) \quad : x = L, t > 0 \quad (3.1c)$$

where T_{m1} and T_{m2} are mean temperature of oscillation, T_0 and T_L are amplitude of thermal wave, Φ_0 and Φ_L are the phase angles at the both the ends of the specimen respectively.

The differential equation and its boundary conditions can be simplified by using temperature difference variable $\theta = T - T_a$. It reduces to

$$\frac{\partial^2 \theta}{\partial x^2} = \frac{1}{\alpha} \frac{\partial \theta}{\partial t} + \frac{2h}{rk} \theta \quad (3.2)$$

$$\theta = 0 \quad t=0, \quad \text{for } 0 \leq x \leq L \quad (3.2a)$$

$$\theta = \theta_{m1} + T_0 \exp(i(\omega t + \Phi_0)) \quad : \quad x = 0, t > 0 \quad (3.2b)$$

$$\theta = \theta_{m2} + T_L \exp(i(\omega t + \Phi_L)) \quad : \quad x = L, t > 0 \quad (3.2c)$$

3.2.1 Method of solution

This type of boundary value problems is not solvable by method of variable separation because the boundary conditions are not separable due to its non-homogeneous [2] nature. These equations can be solved analytically either by splitting boundary condition method or by using Laplace transform technique. Both the solution methods lead to same solution but it is a matter of convenience to use a particular method. In the case of splitting boundary condition method the equations and its boundary conditions are broken into space and space-time dependent equations where the latter can be solved by method of residue or Laplace transform method. In the case of direct Laplace transform method, the partial differential equations are initially converted to total differential in the Laplace plane.

3.2.2 Splitting Boundary Condition Method

In this method, the governing equation and its boundary condition can be splitted [2] to a set of governing equations and its associated boundary conditions, which can be easily solved independently. The individual solutions are superimposed to get the final solution. In the situations where transient terms are required, the splitting boundary conditions do not provide any advantages over the direct Laplace transform. However if steady periodic solution is required the splitting boundary condition method can be employed to get the solution easily.

The fundamental equation, Eq. (3.2) can be decomposed as,

$$\theta = \varphi_{x,t} + \psi_x$$

(3.3) where ψ_x is the solution of the steady state problem and $\varphi_{x,t}$ is the solution of a transient problem. The steady state equation and its boundary conditions can be written as,

$$\frac{d^2 \psi_x}{dx^2} = \frac{2h}{rk} \psi_x \quad (3.4)$$

$$\psi_x = \theta_{m1}, \quad : x = 0, t > 0 \quad (3.4a)$$

$$\psi_x = \theta_{m2}, \quad : x = L, t > 0 \quad (3.4b)$$

The transient equation with its initial and boundary conditions are,

$$\frac{\partial^2 \phi_{x,t}}{\partial x^2} = \frac{1}{\alpha} \frac{\partial \phi_{x,t}}{\partial t} + \frac{2h}{rk} \phi_{x,t} \quad (3.5)$$

$$\phi_{x,t} = T_0 \exp(i(\omega t + \Phi_0)), \quad x = 0, t > 0 \quad (3.5a)$$

$$\phi_{x,t} = T_L \exp(i(\omega t + \Phi_L)), \quad x = L, t > 0 \quad (3.5b)$$

$$\phi_{x,t} = -\psi_x, \quad t = 0, \text{ for } 0 \leq x \leq L \quad (3.5c)$$

The general solution of the Eq. (3.4) is,

$$\psi_x = C_1 \exp(\beta x) + C_2 \exp(-\beta x) \quad (3.6)$$

where $\beta = \sqrt{2h/rk}$. C_1 and C_2 are the arbitrary constants, which are determined by using boundary conditions (3.4a) and (3.4b) as,

$$C_1 = \frac{\theta_{m2} - \theta_{m1} \exp(-\beta L)}{\exp(\beta L) - \exp(-\beta L)} \quad (3.7)$$

$$C_2 = \frac{\theta_{m1} \exp(\beta L) - \theta_{m2}}{\exp(\beta L) - \exp(-\beta L)} \quad (3.8)$$

By substituting the value of C_1 and C_2 in the Eq. (3.6), yields

$$\psi_x = \frac{\theta_{m2} \sinh(\beta x) + \theta_{m1} \sinh(\beta(L - x))}{\sinh(\beta L)} \quad (3.9)$$

Taking the Laplace transform of the Eq. (3.5) and its boundary conditions yields,

$$\frac{d^2 \bar{\phi}}{dx^2} = \frac{1}{\alpha} [s\bar{\phi} - \phi_{t=0}] + \frac{2h}{rk} \bar{\phi} \quad (3.10)$$

$$\bar{\phi} = \frac{T_0 \exp(i\Phi_0)}{(s - i\omega)} \quad : x = 0, t > 0 \quad (3.10a)$$

$$\bar{\phi} = \frac{T_L \exp(i\Phi_L)}{(s - i\omega)} \quad : x = L, t > 0 \quad (3.10b)$$

The Eq. (3.10) can be rewritten by using Eq. (3.5c) as,

$$\left[\frac{d^2}{dx^2} - \left(\frac{s}{\alpha} + \frac{2h}{rk} \right) \right] \bar{\phi} = \frac{\psi_x}{\alpha} \quad (3.11)$$

Solution of this differential equation has complementary function (C.F.) and particular integral (P.I.), which are stated as,

$$\bar{\varphi} = \overbrace{C_3 \exp(m_1 x) + C_4 \exp(-m_1 x)}^{\text{C.F.}} - \overbrace{\frac{\theta_{m2} \sinh(\beta x) + \theta_{m1} \sinh(\beta(L-x))}{s \sinh(\beta L)}}^{\text{P.I.}} \quad (3.12)$$

$$\text{where } m_1 = \sqrt{\frac{s}{\alpha} + \frac{2h}{rk}} \quad (3.13)$$

Use of first boundary condition given by Eq. (3.10a), yields,

$$\frac{T_0 \exp(\Phi_0)}{(s - i\omega)} = C_3 + C_4 - \frac{\theta_{m1}}{s} \quad (3.14)$$

And applying second boundary condition given by Eq. (3.10b) yields,

$$\frac{T_L \exp(i\Phi_L)}{(s - i\omega)} = C_3 \exp(m_1 L) + C_4 \exp(-m_1 L) - \frac{\theta_{m2}}{s} \quad (3.15)$$

The expressions for C_3 and C_4 can be obtained from Eqs. (3.14) and (3.15) as,

$$C_3 = \frac{\left(\frac{\theta_{m2}}{s} + \frac{T_L \exp(i\Phi_L)}{(s - i\omega)} \right) - \left(\frac{\theta_{m1}}{s} + \frac{T_0 \exp(i\Phi_0)}{(s - i\omega)} \right) \exp(-m_1 L)}{\exp(m_1 L) - \exp(-m_1 L)} \quad (3.16)$$

$$C_4 = \frac{\left(\frac{\theta_{m1}}{s} + \frac{T_0 \exp(i\Phi_0)}{(s - i\omega)} \right) \exp(m_1 L) - \left(\frac{\theta_{m2}}{s} + \frac{T_L \exp(i\Phi_L)}{(s - i\omega)} \right)}{\exp(m_1 L) - \exp(-m_1 L)} \quad (3.17)$$

Substituting the values of C_3 and C_4 in Eq. (3.12) yields,

$$\begin{aligned} \bar{\varphi} = & \frac{\theta_{m1} \sinh(m_1(L-x))}{s \sinh(m_1 L)} + \frac{\theta_{m2} \sinh(m_1 L)}{s \sinh(m_1 L)} + \frac{T_0 \exp(i\Phi_0) \sinh(m_1(L-x))}{(s - i\omega) \sinh(m_1 L)} \\ & + \frac{T_L \exp(i\Phi_L) \sinh(m_1 L)}{(s - i\omega) \sinh(m_1 L)} - \frac{\theta_{m2} \sinh(\beta x) + \theta_{m1} \sinh(\beta(L-x))}{s \sinh(\beta L)} \end{aligned} \quad (3.18)$$

The Eq. (3.18) consists of five terms in Laplace domain. The time domain solution is obtained by taking Laplace inversion of these five terms.

The inverse transform of the first four terms are obtained by the method of residues and the inverse of the last term is readily available in Laplace table [147]. For the function $F(x)$ and its Laplace transform $F(s)$, are related by contour integral and residues,

$$F(x) = \frac{1}{2\pi i} \int_{y-i\infty}^{y+i\infty} e^{st} F(s) ds = \Sigma \text{ Residue of } e^{st} F(s) \quad (3.19)$$

Inverse transform of first term:

The poles of the integrand function of the first term are given by $s = 0$

and $\sqrt{(s/\alpha) + (2h/rk)} L = n \pi i$ for $n = 1, 2, \dots$.

Thus the poles are $s = 0$

and $s = s_n = -\left\{(n\pi/L)^2 + (2h/rk)\right\}\alpha$ (3.20)

Residue of the first term is,

$$\begin{aligned} \text{Res(I)} &= \lim_{s \rightarrow 0} \left[\frac{\theta_{m1} s \exp(st) \sinh(m_1(L-x))}{s \sinh(m_1 L)} \right] \\ &+ \lim_{s \rightarrow s_n} \left[\frac{\theta_{m1} (s - s_n) \exp(st) \sinh(m_1(L-x))}{s \sinh(m_1 L)} \right] \end{aligned} \quad (3.21)$$

The second term of the above expression is indeterminate form, this residue can be determined as,

$$\begin{aligned} \text{Res(I)} &= \frac{\theta_{m1} \sinh \beta(L-x)}{\sinh(\beta L)} \\ &+ \theta_{m1} \lim_{s \rightarrow s_n} \left[\frac{\partial(s - s_n)/\partial s}{\partial \{\sinh(m_1 L)\}/\partial s} \right] \lim_{s \rightarrow s_n} \left[\frac{\exp(s_n t) \sinh m_1(L-x)}{s} \right] \end{aligned} \quad (3.22)$$

On differentiating numerator and denominators with respect to s , results the inverse transform of the first term as,

$$\text{Res(I)} = \frac{\theta_{m1} \sinh \beta(L-x)}{\sinh(\beta L)} + \theta_{m1} \sum_{n=1}^{\infty} \frac{2\alpha n \pi i \exp(s_n t) \sinh[n\pi i(1-\varepsilon)]}{L^2 s_n \cosh(n\pi i)} \quad (3.23)$$

Inverse transform of second term:

Similar to the first term, the inverse transform of second term is,

$$\text{Res(II)} = \frac{\theta_{m2} \sinh(\beta x)}{\sinh(\beta L)} + \theta_{m2} \sum_{n=1}^{\infty} \frac{2\alpha n \pi i \exp(s_n t) \sinh[n\pi i \varepsilon]}{L^2 s_n \cosh(n\pi i)} \quad (3.24)$$

Inverse transform of third term:

The poles of the integrand function of the third term are given by $s = i \omega$

and $\sqrt{(s/\alpha) + (2h/rk)} L = n \pi i$, for $n = 1, 2, 3, \dots$.

Thus the poles are, $s = i \omega$

and $s = s_n = -\left\{(n\pi/L)^2 + (2h/rk)\right\}\alpha$ (3.25)

The residue is given as,

$$\begin{aligned} \text{Res(III)} = & \lim_{s \rightarrow i\omega} \left[\frac{T_0 \exp(i\Phi_0)(s - i\omega) \exp(st) \sinh m_1 (L - x)}{(s - i\omega) \sinh(m_1 L)} \right] \\ & + \lim_{s \rightarrow s_n} \left[\frac{T_0 \exp(i\Phi_0)(s - s_n) \exp(st) \sinh m_1 (L - x)}{(s - i\omega) \sinh(m_1 L)} \right] \end{aligned} \quad (3.26)$$

On further simplifying, the above equation yields,

$$\begin{aligned} \text{Res(III)} = & T_0 \exp(i(\omega t + \Phi_0)) \frac{\sinh(\beta_1 (L - x))}{\sinh(\beta_1 L)} \\ & + T_0 \exp(i\Phi_0) \sum_{n=1}^{\infty} \frac{2\alpha n \pi i \exp(s_n t) \sinh[n\pi i(1 - \varepsilon)]}{L^2 (s_n - i\omega) \cosh(n\pi i)} \end{aligned} \quad (3.27)$$

where $\beta_1 = \sqrt{\frac{i\omega}{\alpha} + \frac{2h}{rk}}$ (3.28)

Inverse transform of fourth term:

Similar to the third term, the inverse transform of forth term is

$$\begin{aligned} \text{Res(IV)} = & T_L \exp(i(\omega t + \Phi_L)) \frac{\sinh(\beta_1 x)}{\sinh(\beta_1 L)} \\ & + T_L \exp(i\Phi_L) \sum_{n=1}^{\infty} \frac{2\alpha n \pi i \exp(s_n t) \sinh(n\pi i\varepsilon)}{L^2 (s_n - i\omega) \cosh(n\pi i)} \end{aligned} \quad (3.29)$$

The Laplace inversion of the fifth term can be obtained from a standard Laplace transform table [147] as,

$$\text{Fifth term} = - \frac{\theta_{m2} \sinh(\beta x) + \theta_{m1} \sinh(\beta(L - x))}{\sinh(\beta L)} \quad (3.30)$$

The Eq. (3.3) can be written as the summation of Equations (3.9) and time domain solution of Eq. (3.18). Thus the combinations of Eq. (3.9), (3.23), (3.24), (3.27), (3.29), and (3.30) represents solution of Eq. (3.2). It may be noted that the solution expressed by ψ_x in Eq. (3.9) get cancelled with Eq. (3.30). Thus the final solution can be expressed as in two parts, steady periodic and the transient part as given by Equations (3.31) and (3.32) respectively as,

$$\begin{aligned} \theta_s = & \frac{\theta_{m1} \sinh(\beta(L - x))}{\sinh(\beta L)} + \frac{\theta_{m2} \sinh(\beta x)}{\sinh(\beta L)} + T_0 \exp(i(\omega t + \Phi_0)) \frac{\sinh(\beta_1 (L - x))}{\sinh(\beta_1 L)} \\ & + T_L \exp(i(\omega t + \Phi_L)) \frac{\sinh(\beta_1 x)}{\sinh(\beta_1 L)} \end{aligned} \quad (3.31)$$

$$\theta_T = \sum_{n=1}^{\infty} \frac{\exp(s_n t) [\theta_{m1} \sinh\{n\pi i (1-\varepsilon)\} + \theta_{m2} \sinh(n\pi i\varepsilon)] 2\alpha n \pi i}{L^2 s_n \cosh(n\pi i)} + \sum_{n=1}^{\infty} \frac{\exp(s_n t) [T_0 \exp(i\Phi_0) \sinh\{n\pi i (1-\varepsilon)\} + T_L \exp(i\Phi_L) \sinh(n\pi i\varepsilon)] 2\alpha n \pi i}{L^2 (s_n - i\omega) \cosh(n\pi i)} \quad (3.32)$$

3.2.3 Direct Laplace Transform Method

The problem defined by Eq. (3.2) can also be solved directly by using Laplace transform technique without splitting the boundary conditions. The employment of the Laplace transform in parabolic heat conduction equation leads to a second order differential equation in the spatial variable, and this resulting differential equation is then analytically solved. The inversion of the solution leads to the final solution in time domain.

Taking the Laplace transform of the Eq. (3.2) and its boundary conditions yields,

$$\frac{d^2 \bar{\theta}}{dx^2} = \frac{1}{\alpha} [s\bar{\theta} - \theta_{t=0}] + \frac{2h}{rk} \bar{\theta} \quad (3.33)$$

$$\bar{\theta} = \frac{\theta_{m1}}{s} + \frac{T_0 \exp(i\Phi_0)}{(s - i\omega)} \quad : \quad x=0, t > 0 \quad (3.33a)$$

$$\bar{\theta} = \frac{\theta_{m2}}{s} + \frac{T_L \exp(i\Phi_L)}{(s - i\omega)} \quad : \quad x=L, t > 0 \quad (3.33b)$$

The general solution of the Eq. (3.33) is

$$\bar{\theta} = C_5 \exp(m_1 x) + C_6 \exp(-m_1 x) \quad (3.34)$$

The arbitrary constants, C_5 and C_6 are expressed as,

$$C_5 = \frac{\left(\frac{\theta_{m2}}{s} + \frac{T_L \exp(i\Phi_L)}{(s - i\omega)} \right) - \left(\frac{\theta_{m1}}{s} + \frac{T_0 \exp(i\Phi_0)}{(s - i\omega)} \right) \exp(-m_1 L)}{\exp(m_1 L) - \exp(-m_1 L)} \quad (3.35)$$

$$C_6 = \frac{\left(\frac{\theta_{m1}}{s} + \frac{T_0 \exp(i\Phi_0)}{(s - i\omega)} \right) \exp(m_1 L) - \left(\frac{\theta_{m2}}{s} + \frac{T_L \exp(i\Phi_L)}{(s - i\omega)} \right)}{\exp(m_1 L) - \exp(-m_1 L)} \quad (3.36)$$

Substituting the value of constants C_5 and C_6 into Eq. (3.34) yields,

$$\bar{\theta} = \frac{\left[\frac{\theta_{m1}}{s} + \frac{T_0 \exp(i\Phi_0)}{(s - i\omega)} \right] [\exp(m_1(L - x)) - \exp(-m_1(L - x))]}{\exp(m_1 L) - \exp(-m_1 L)} + \frac{\left[\frac{\theta_{m2}}{s} + \frac{T_L \exp(i\Phi_L)}{(s - i\omega)} \right] [\exp(m_1 x) - \exp(-m_1 x)]}{\exp(m_1 L) - \exp(-m_1 L)} \quad (3.37)$$

The above equation can be written in hyperbolic functions as,

$$\bar{\theta} = \frac{\theta_{m1} \sinh(m_1(L - x))}{s \sinh(m_1 L)} + \frac{\theta_{m2} \sinh(m_1 x)}{s \sinh(m_1 L)} + \frac{T_0 \exp(i\Phi_0) \sinh(m_1(L - x))}{(s - i\omega) \sinh(m_1 L)} + \frac{T_L \exp(i\Phi_L) \sinh(m_1 x)}{(s - i\omega) \sinh(m_1 L)} \quad (3.38)$$

This is the solution in Laplace domain. The original time domain solution is obtained by taking inverse transform of Eq. (3.38). Therefore terms are same as the first four terms of the Eq. (3.18). It may be related that the fifth term in Eq. (3.18) is cancelled with the Eq. (3.9) to yield the solution of the original Eq. (3.3). Hence the solution by direct Laplace transform is same as that of splitting boundary condition described by the last section in Equations (3.31) and (3.32).

3.3 Derivation of Parameters for the Measurement of the Thermophysical Properties

3.3.1 Exact Solution

For large value of time all transient disturbances fade away and the remaining steady periodic solution given by Eq. (3.31) is required to measure the amplitude ratio and phase change. The Eq. (3.31) can be further simplified for equal amplitudes and phases on both sides of the sample to yield the complex steady periodic part as,

$$\theta_s = \frac{T_0 \exp(i(\omega t + \Phi_0)) \cosh(\beta_1(L - 2x)/2)}{\cosh(\beta_1 L/2)} \quad (3.39)$$

The ratio of the complex amplitude at any point x and at $x = 0$ is given by,

$$A_z = \frac{\cos(\kappa(L - 2x)/2)}{\cos(\kappa L/2)} \quad (3.40)$$

where κ is the wave number defined as,

$$\kappa = \sqrt{(\omega/2\alpha) + (h/rk i)} (1 - i) \quad (3.41)$$

Complex amplitude ratio given by Eq. (3.40) varies from unity to zero with increase in x from zero to $x = L$ ($L \rightarrow \infty$). The Eq. (3.40) can be expressed as,

$$A_z = A \exp(-i\Phi) \quad (3.42)$$

where the amplitude A and phase Φ can be expressed as

$$A = \sqrt{\text{Re}[A_z]^2 + \text{Im}[A_z]^2} \quad (3.43)$$

$$\Phi = \arctan\left(\frac{\text{Im}[A_z]}{\text{Re}[A_z]}\right) \quad (3.44)$$

Eq. (3.41) can also be written as

$$\kappa = \left\{ (-h/rk) + \left[(h/rk)^2 + (\omega/2\alpha)^2 \right]^{1/2} \right\}^{1/2} - i \left\{ (h/rk) + \left[(h/rk)^2 + (\omega/2\alpha)^2 \right]^{1/2} \right\}^{1/2} \quad (3.45)$$

The quantity $\omega/2\alpha$ corresponds to the heat conduction and h/rk to the thermal losses from the surface. Defining a quantity δ as the half of the ratio of thermal loss to heat conductance as,

$$\delta = \frac{\alpha h}{\omega rk} \quad (3.46)$$

The Eq. (3.45) can be written as,

$$\kappa = \left(\frac{\omega}{2\alpha} \right)^{1/2} \left\{ \left[-2\delta + \left\{ (2\delta)^2 + 1 \right\}^{1/2} \right]^{1/2} - i \left[2\delta + \left\{ (2\delta)^2 + 1 \right\}^{1/2} \right]^{1/2} \right\} \quad (3.47)$$

Using, $2\delta = \sinh(g)$, the Eq. (3.47) can be further reduced to

$$\kappa = \left(\frac{\omega}{2\alpha} \right)^{1/2} \left\{ \left[-\sinh g + \cosh g \right]^{1/2} - i \left[\sinh g + \cosh g \right]^{1/2} \right\} \quad (3.48)$$

On simplifying and rearranging the terms

$$\kappa = (\omega/2\alpha)^{1/2} [(1 - f) - i(1 + f)] \quad (3.49)$$

$$\text{where} \quad f = \sinh\left((\sinh^{-1} 2\delta)/2\right) \quad (3.50)$$

Introducing dimensionless parameters

$$\gamma = L\sqrt{\omega/2\alpha} \quad (3.51)$$

$$\varepsilon = x/L \quad (3.52)$$

and substituting the value of κ into Eq. (3.40) yields

$$A_Z = \frac{\cos\left(\frac{\{(1-f) - i(1+f)\}\gamma(1-2\varepsilon)}{2}\right)}{\cos\left(\frac{\{(1-f) - i(1+f)\}\gamma}{2}\right)} \quad (3.53)$$

$$\begin{aligned} \text{Defining,} \quad C &= \frac{\gamma}{2}(1-2\varepsilon)(1-f), & E &= \frac{\gamma}{2}(1-2\varepsilon)(1+f), \\ F &= \frac{\gamma}{2}(1-f), & G &= \frac{\gamma}{2}(1+f) \end{aligned} \quad (3.54)$$

A_Z can be written as

$$A_Z = \frac{\cos(C - iE)}{\cos(F - iG)} \quad (3.55)$$

By separation of real and imaginary part, the real amplitude ratio and phase shift can be expressed as

$$A = \sqrt{\frac{\sinh^2 E + \cos^2 C}{\cos^2 F + \sinh^2 G}} \quad (3.56)$$

$$\text{and} \quad \Phi = \tan^{-1} \frac{\tan F \tanh G - \tan C \tanh E}{1 + \tan C \tanh E \tan F \tanh G} \quad (3.57)$$

It can be seen from the equations the amplitude ratio and the phase shift depend only on the two variables γ and f with fixed value of ε . So, at a fixed location of ε , the amplitude ratio and phase shift can be evaluated from the measured temperatures oscillation. The measurement of the phase difference and the amplitude ratio allows the measurement two properties such as α , h from the Eqs. (3.56) and (3.57) respectively.

3.3.2 Approximate Solution

In many of such measurement systems, the heat quantity $h/r\lambda$ is small compared to heat conduction quantity $\omega/2\alpha$. In such cases $\delta \ll 1$. Thus Eq. (3.47) reduced to

$$\kappa = (\omega/2\alpha)^{\frac{1}{2}} \left\{ (1-2\delta)^{\frac{1}{2}} - i(1+2\delta)^{\frac{1}{2}} \right\} \quad (3.58)$$

By expansion and neglecting higher order terms, Eq. (3.58) yields,

$$\kappa = \left(\frac{\omega}{2\alpha} \right)^{\frac{1}{2}} [(1-\delta) - i(1+\delta)] \quad (3.59)$$

Substituting the value of κ into Eq. (3.40) yields,

$$A_Z = \frac{\cos\left(\frac{\{(1-\delta) - i(1+\delta)\}\gamma(1-\varepsilon)}{2}\right)}{\cos\left(\frac{\{(1-\delta) - i(1+\delta)\}\gamma}{2}\right)} \quad (3.60)$$

Defining, $C = \frac{\gamma}{2}(1-2\varepsilon)(1-\delta)$, $E = \frac{\gamma}{2}(1-2\varepsilon)(1+\delta)$,

$$F = \frac{\gamma}{2}(1-\delta), \quad G = \frac{\gamma}{2}(1+\delta) \quad (3.61)$$

Eq. (3.60) can be written as,

$$A_Z = \frac{\cos(C - iE)}{\cos(F - iG)} \quad (3.62)$$

By separation of real and imaginary part, the real amplitude ratio and phase shift can be expressed as,

$$A = \sqrt{\frac{\sinh^2 E + \cos^2 C}{\cos^2 F + \sinh^2 G}} \quad (3.63)$$

and

$$\Phi = \tan^{-1} \frac{\tan F \tanh G - \tan C \tanh E}{1 + \tan C \tanh E \tan F \tanh G} \quad (3.64)$$

3.4 General Solution of Differential Equation without Lateral Heat Transfer

In many situations the measurements are carried out on the sample where the convective and radiative heat losses are negligible, this results an adiabatic surface condition. The solution of the resultant governing equations can either be derived from the previous expression given in section (3.2.3) or can be derived directly. Since, the splitting boundary conditions is not applicable for this type of governing differential equation, the direct Laplace transform technique seems to be a prominent option. In this section the important steps for the derivation is highlighted.

The governing differential equation can be reduced from Eq. (3.2) as,

$$\frac{\partial^2 \theta}{\partial x^2} = \frac{1}{\alpha} \frac{\partial \theta}{\partial t} \quad (3.65)$$

$$\theta = 0 \quad t = 0, \text{ for } 0 \leq x \leq L \quad (3.65a)$$

$$\theta = \theta_{m1} + T_0 \exp(i(\omega t + \Phi_0)) \quad : x = 0, t > 0 \quad (3.65b)$$

$$\theta = \theta_{m2} + T_L \exp(i(\omega t + \Phi_L)) \quad : x = L, t > 0 \quad (3.65c)$$

The Laplace transform of Eq. (3.65) along with its initial and boundary conditions can be expressed as,

$$\frac{d^2 \bar{\theta}}{dx^2} = \frac{1}{\alpha} [s \bar{\theta} - \theta_{t=0}] \quad (3.66)$$

$$\bar{\theta} = \frac{\theta_{m1}}{s} + \frac{T_0 \exp(i\Phi_0)}{(s - i\omega)} \quad : x = 0, t > 0 \quad (3.66a)$$

$$\bar{\theta} = \frac{\theta_{m2}}{s} + \frac{T_L \exp(i\Phi_L)}{(s - i\omega)} \quad : x = L, t > 0 \quad (3.66b)$$

Since the general solution of the Eq. (3.66) is

$$\bar{\theta} = C_7 \exp(m_2 x) + C_8 \exp(-m_2 x) \quad (3.67)$$

$$\text{where } m_2 = \sqrt{(s/\alpha)} \quad (3.68)$$

The arbitrary constants are C_7 and C_8 which can be determined from the boundary conditions (3.66a) and (3.66b) respectively.

It may be noted that the solution of Eq. (3.67) is same as that of Eq. (3.38) in Laplace domain when m_1 is replaced by m_2 . Similarly, the solution in time domain can be derived from Equations (3.31) and (3.32) by replacing β_1 by β_2 and $\beta \rightarrow 0$. The definition of relevant variables is

$$\beta_2 = \sqrt{i\omega/\alpha} \quad \text{and} \quad s_n = -\left\{ (n\pi/L)^2 \alpha \right\} \quad (3.69)$$

With the above variables the solution of Eq. (3.65) in steady periodic part and transient part can be expressed as,

$$\theta = \theta_s + \theta_T \quad (3.70)$$

Steady part of θ (i.e. θ_s) is,

$$\begin{aligned} \theta_s = & \frac{\theta_{m1}(L-x)}{L} + \frac{\theta_{m2} x}{L} + T_0 \exp(i(\omega t + \Phi_0)) \frac{\sinh \beta_2 (L-x)}{\sinh(\beta_2 L)} \\ & + T_L \exp(i(\omega t + \Phi_L)) \frac{\sinh(\beta_2 x)}{\sinh(\beta_2 L)} \end{aligned} \quad (3.71)$$

Transient part of θ (i.e. θ_T) is,

$$\theta_T = \sum_{n=1}^{\infty} \frac{\exp(s_n t) [\theta_{m1} \sinh\{n\pi i (1-\varepsilon)\} + \theta_{m2} \sinh(n\pi i \varepsilon)] 2\alpha n \pi i}{L^2 (s_n) \cosh(n\pi i)} + \sum_{n=1}^{\infty} \frac{\exp(s_n t) [T_0 \exp(i\Phi_0) \sinh\{n\pi i (1-\varepsilon)\} + T_L \exp(i\Phi_L) \sinh(n\pi i \varepsilon)] 2\alpha n \pi i}{L^2 (s_n - i\omega) \cosh(n\pi i)} \quad (3.72)$$

For a large value of time all transient disturbances given by Eq. (3.72) fade away and only the remaining steady periodic solutions are required to measure the amplitude ratio and phase change. Eq. (3.71) is further simplified by applying same amplitudes and phases on both sides of the rod. Complex steady periodic solution yields

$$\theta_s = \theta_{m1} + \frac{T_0 \exp(i(\omega t + \Phi_0)) \cosh(\beta_2 (L - 2x)/2)}{\cosh(\beta_2 L/2)} \quad (3.73)$$

Under steady state condition, the signal temperature consists of mean temperature θ_{m1} and a periodically oscillating temperature as stated by Eq. (3.73).

The complex amplitude ratio between any point on the specimen and at the surface $x = 0$ is given by the following equation.

$$A_z = \frac{\cos(\kappa (L - 2x)/2)}{\cos(\kappa L/2)} \quad (3.74)$$

where κ is the wave number given by

$$\kappa = \sqrt{\omega/2\alpha}(1-i) \quad (3.75)$$

Following the similar procedure as described by Equations (3.42) to (3.55) the real part of amplitude ratio and phase shift can be expressed as,

$$A = \sqrt{\frac{\sinh^2 C + \cos^2 C}{\cos^2 F + \sinh^2 F}} \quad (3.76)$$

$$\text{and} \quad \Phi = \tan^{-1} \frac{\tan F \tanh F - \tan C \tanh C}{1 + \tan C \tanh C \tan F \tanh F}$$

(3.77)

In absence of lateral heat transfer the variables can be defined as,

$$C = \frac{\gamma}{2}(1-2\varepsilon), \quad F = \frac{\gamma}{2}, \quad \text{and} \quad \gamma = L(\omega/2\alpha)^{1/2} \quad (3.78)$$

It can be seen from the above Equations (3.76) and (3.77) that for a fixed value of ε , the amplitude and phase difference depend only on one variable γ .

Thus measurement of γ either from amplitude ratio or phase shift yields the measurement of α .

3.5 Deduction of Solutions for Specific Boundary Conditions

It is found in literature that the different investigators have proposed different steady periodic solutions depending on the boundary conditions. Their solutions are readily obtained from generalized steady periodic solutions, which are summarized below;

Case I

The solution for a rod, (0-L) having adiabatic boundary [5] condition at $x=L$ is obtained by substituting $2L$ in place of L in generalized steady periodic solution given by Eq. (3.31).

$$\theta_s = \frac{\theta_{m1} \sinh(\beta(2L-x))}{\sinh(2\beta L)} + \frac{\theta_{m2} \sinh(\beta x)}{\sinh(2\beta L)} + T_0 \exp(i(\omega t + \Phi_0)) \frac{\sinh(\beta_1(2L-x))}{\sinh(2\beta_1 L)} + T_L \exp(i(\omega t + \Phi_L)) \frac{\sinh(\beta_1 x)}{\sinh(2\beta_1 L)} \quad (3.79)$$

Using symmetrical boundary condition, it can be simplified as,

$$\theta_s = \frac{\theta_{m1} \cosh(\beta(L-x))}{\cosh(\beta L)} + T_0 \exp(i(\omega t + \Phi_0)) \frac{\cosh(\beta_1(L-x))}{\cosh(\beta_1 L)} \quad (3.80)$$

If there is no lateral heat loss, Eq. (3.80) reduces to

$$\theta_s = \theta_{m1} + T_0 \exp(i(\omega t + \Phi_0)) \frac{\cosh(\beta_2(L-x))}{\cosh(\beta_2 L)} \quad (3.81)$$

Case II

This derivation represents the solution for a finite sample when one end is subjected to sinusoidal boundary condition and the other end of the rod is connected to a heat sink maintained at constant temperature as obtained by Tomokiyo and Okada [4].

In Eq. (3.31), by substituting $T_L \exp(i(\omega t + \Phi_L))$ equal to zero then equation becomes

$$\theta_s = \frac{\theta_{m1} \sinh \beta(L-x)}{\sinh(\beta L)} + \frac{\theta_{m2} \sinh(\beta x)}{\sinh(\beta L)} + \frac{T_0 \exp(i(\omega t + \Phi_0)) \sinh \beta_1(L-x)}{\sinh(\beta_1 L)} \quad (3.82)$$

This is the solution for a finite sample when one end is subjected to sinusoidal boundary and other end of the rod connected to a heat sink maintained constant temperature.

Case III

Angstrom's result, for long sample, where one end of the rod is subjected to a sinusoidal temperature variation and other end tends to infinite can be derived from Eq. (3.31) with $L \rightarrow \infty$.

The generalized steady periodic solution given by Eq. (3.31) of transient heat conduction can also be written as,

$$\begin{aligned}\theta_s = & \frac{\theta_{m1}[\exp(\beta(L-x)) - \exp(-\beta(L-x))]}{\exp(\beta L) - \exp(-\beta L)} + \frac{\theta_{m2}[\exp(\beta x) - \exp(-\beta x)]}{\exp(\beta L) - \exp(-\beta L)} \\ & + \frac{T_0 \exp(i(\omega t + \Phi_0))[\exp(\beta_1(L-x)) - \exp(-\beta_1(L-x))]}{\exp(\beta_1 L) - \exp(-\beta_1 L)} \\ & + \frac{T_L \exp(i(\omega t + \Phi_L))[\exp(\beta_1 x) - \exp(-\beta_1 x)]}{\exp(\beta_1 L) - \exp(-\beta_1 L)}\end{aligned}\quad (3.83)$$

If length of the bar tends to infinity (long samples) then second and fourth terms of the Eq. (3.83) becomes zero and remaining terms are further simplified as,

$$\begin{aligned}\theta_s = & \frac{\theta_{m1}[\exp(\beta(L-x)) - \exp(-\beta(L-x))]}{\exp(\beta L) - \exp(-\beta L)} \\ & + \frac{T_0 \exp(i(\omega t + \Phi_0))[\exp(\beta_1(L-x)) - \exp(-\beta_1(L-x))]}{\exp(\beta_1 L) - \exp(-\beta_1 L)}\end{aligned}\quad (3.84)$$

It can also be written as

$$\begin{aligned}\theta_s = & \theta_{m1} \left[\frac{\exp(\beta L) \exp(-\beta x)}{\exp(\beta L) - \exp(-\beta L)} - \frac{\exp(-\beta(L-x))}{\exp(\beta L) - \exp(-\beta L)} \right] \\ & + T_0 \exp(i(\omega t + \Phi_0)) \left[\frac{\exp(\beta_1 L) \exp(-\beta_1 x)}{\exp(\beta_1 L) - \exp(-\beta_1 L)} - \frac{\exp(-\beta_1(L-x))}{\exp(\beta_1 L) - \exp(-\beta_1 L)} \right]\end{aligned}\quad (3.85)$$

On the limiting conditions of $L \rightarrow \infty$, Eq. (3.85) reduces to

$$\theta_s = \theta_{m1} \exp(-\beta x) + T_0 \exp(i(\omega t + \Phi_0)) \exp(-\beta_1 x) \quad (3.86)$$

If there is no lateral heat loss, then Eq. (3.86) reduces to

$$\theta_s = \theta_{m1} + T_0 \exp(i(\omega t + \Phi_0)) \exp(-\beta_2 x) \quad (3.87)$$

3.6 Effect of Settling Time on Measurement

In the transient technique, the time to achieve the pseudo-steady state is an important factor known as settling time [148,149]. This factor determines, the elapsed time required to attain the steady state. For high thermal conductivity materials where the attenuation is fast enough, the settling time is very small (i.e., order of fraction of a second). At the other extreme, for low thermal conductivity materials, it may be of hours.

A near steady state condition is assumed when time required for the temperature of the transient part to achieve 2% of the steady state temperature. This section describes the procedure to approximately estimate the settling time.

3.6.1 Settling Time with Lateral Heat Transfer

The Eq. (3.32) can be simplified for same amplitude and phase on both sides of the sample as,

$$\theta_T = T_0 \exp(i\Phi_0) \sum_{n=1}^{\infty} \frac{4n\pi\alpha i^2 \sin(n\pi/2) \cos\{n\pi(1-2\varepsilon)/2\} \exp(s_n t)}{L^2 (s_n - i\omega) \cosh(n\pi i)} \quad (3.88)$$

The transient response for unit input can be expressed as,

$$I(\theta_T) = \exp(-i\omega t) \sum_{n=1}^{\infty} \frac{4n\pi\alpha i^2 \sin(n\pi/2) \cos\{n\pi(1-2\varepsilon)/2\} \exp(s_n t)}{L^2 (s_n - i\omega) \cosh(n\pi i)} \quad (3.89)$$

The response of the signal decreases exponentially. The higher harmonics of the series decrease much faster than the fundamental, which persists for a longer time after the initial disturbance. Thus the contribution of the Eq. (3.89) may be approximated due to the first term of the series as,

$$I(\theta_T) = \exp(-i\omega t) \frac{4\pi\alpha \cos\{\pi(1-2\varepsilon)/2\} \exp(s_1 t)}{L^2 (s_1 - i\omega)} \quad (3.90)$$

Where $\cosh(n\pi i) = (-1)^n$

The real and imaginary part of this response can be written by the following two equations.

$$\text{Re}[I(\theta_T)] = \frac{4\pi\alpha \cos\{\pi(1-2\varepsilon)/2\} \exp(s_1 t) (\omega \sin \omega t + s_1 \cos \omega t)}{L^2 (s_1^2 + \omega^2)} \quad (3.91)$$

$$\text{Im}[I(\theta_T)] = \frac{4\pi\alpha \cos\{\pi(1-2\varepsilon)/2\} \exp(s_1 t) (\omega \cos \omega t - s_1 \sin \omega t)}{L^2 (s_1^2 + \omega^2)} \quad (3.92)$$

Thus the magnitude of the temperature amplitude in the transient Eq. (3.90) is given by $(\theta_T)_R = \sqrt{\{[\text{Re}[I(\theta_T)]]\}^2 + \{[\text{Im}[I(\theta_T)]]\}^2}$. This can be expressed as,

$$(\theta_T)_R = \frac{4\pi\alpha \sin(\pi/2) \cos\{\pi(1-2\varepsilon)/2\} \exp\left(-\frac{\alpha t}{L^2} \pi^2 - \frac{2h}{rk} \alpha t\right)}{L^2 \sqrt{\left(\frac{\alpha}{L^2} \pi^2 + \frac{2h}{rk} \alpha\right)^2 + \omega^2}} \quad (3.93)$$

Introducing dimensionless parameter $\varsigma = \alpha t/L^2$, and use of quantities δ , γ and ε in Eq. (3.93) yields,

$$(\theta_T)_R = \frac{4\pi \cos[\pi(1-2\varepsilon)/2] \exp[\varsigma(-\pi^2 - 4\gamma^2\delta)]}{\sqrt{(\pi^2 + 4\gamma^2\delta)^2 + (2\gamma^2)^2}} \quad (3.94)$$

Temperature amplitude of transient part = 2% of amplitude of steady part
As per the condition, the equation for the settling time can be expressed as,

$$\varsigma_s = \frac{\text{Ln}(0.02A/b)}{-\pi^2 - 4\gamma^2\delta} \quad (3.95)$$

where
$$b = \frac{4\pi \cos[\pi(1-2\varepsilon)/2]}{\sqrt{(\pi^2 + 4\gamma^2\delta)^2 + (2\gamma^2)^2}} \quad (3.96)$$

The values of ς_s have been computed for the various values of the parameters γ , ε and δ . A graphical presentation of the settling time has been shown in Figs. (4.8) to (4.10).

3.6.2 Settling Time without Lateral Heat Transfer

The settling time expression as given by Eq. (3.95) can be further simplified by substituting $\delta = 0$. Without lateral heat transfer the Eq. (3.95) reduces to

$$\varsigma_s = \frac{\text{Ln}(0.02A/b)}{-\pi^2} \quad (3.97)$$

where
$$b = \frac{4\pi \cos[\pi(1-2\varepsilon)/2]}{\sqrt{(\pi^2)^2 + (2\gamma^2)^2}} \quad (3.98)$$

3.7 Discussions

Theories of transient heat conduction equation with periodic boundary conditions have been presented based on the principle of temperature oscillation

technique. In this principle, thermophysical properties of the specimen have been estimated by the measurement of amplitude attenuation and phase shift. The governing partial differential equation is formulated for physical model of a bar with surface heat loss in one-dimensional form. This equation has been solved for circular geometry of finite length subjected to sinusoidal temperature oscillation at both the ends. The solution of transient conduction problems consists of both steady, periodic and transient decaying parts. Therefore, a complete solution of this problem is derived in the present work. For the general applicability, the equations are presented in dimensionless parameters for the both dependent and independent parameter. The same governing equation is solved by splitting boundary condition method and direct Laplace transform method to show the correctness of the derivation. Also in situation where only steady state solutions are required, the splitting boundary condition method provides a direct and easy method of solution procedure. Different investigators have taken different boundary conditions for the governing equation to derive the solution. A generalized solution, which accommodates a wide range of boundary conditions for the general governing equation, has been derived. The solution for many specific boundary conditions can be derived from the generalized solution. The output response of amplitude (A) and phase shift (ϕ) for the input signal consisting of same amplitude and phase at the both ends of a finite sample length has been computed for various values of ω , γ and $\tilde{\epsilon}$. The computed values are shown graphically for analysis.

For no lateral heat loss ($\tilde{\epsilon} = 0$) the amplitude and phase are shown in Figs. (3.1) to (3.3). Figures (3.1) and (3.2) show respectively the amplitude and phase variation separately. These two figures are combined in Fig. (3.3). These kinds of graphs are convenient for analyzing the experimental data in evaluation of thermal properties for finite sample length, without lateral heat transfer and oscillations at both the ends. It is observed that for dimensionless parameter $\gamma (= L\sqrt{\omega/2\alpha}) \geq 2.6$, the amplitude curve are sharply attenuated and also densely populated over a short distance (i.e., small value of $\tilde{\epsilon}$). This behavior of finite bar presumes that of a semi-infinite system. It can be further explained that, for a sample with $\gamma \geq 2.6$, the amplitude signal at the middle of the sample is almost zero. A detailed derivation for the semi-infinite system has been given in Chapter 4. The sensitiveness of thermal

diffusivity to amplitude or phase is an important factor in measurement principle. Due to the large change in amplitude it is always preferable to measure the thermal diffusivity from the amplitude signal than from the phase shift signal.

The effect of lateral heat transfer has been shown in Fig. (3.4) and comparison of both, with and without heat transfer has been shown in Fig. (3.5). The nature of plots for heat transfer and without heat transfer are same, but the amplitude attenuation is sharper in the presence of heat transfer. A qualitative presentation with heat transfer shifts to the bottom of the curve, without heat transfer as shown in Fig. (3.5).

A simplified approximate solution for small lateral heat transfer has been derived in the section 3.3.2. The numerical results of exact and approximate solution are compared in Figs. (3.6) and (3.7). It is observed that the deviation is more pronounced for larger values of \tilde{h} . Thus the approximate solution is valid for $\tilde{h} \approx 0$ which is reasonable for small amount of lateral heat transfer.

Under steady state condition, the amplitude ratio or phase change is used to determine the thermophysical properties. The time elapsed to collect the steady state data is approximately determined from the settling time. The plot of settling time is shown in Figs. (3.8) to (3.10). Usually the midpoint of the sample is used for the measurement. The settling time at the mid point of the sample increases with increasing \tilde{h} . It can be further explained that for the same specimen length operating under a constant frequency, the lower thermal diffusivity sample yields a large settling time. The Fig. (3.10) indicates that the transient components settle faster with lateral heat transfer yielding small settling time.

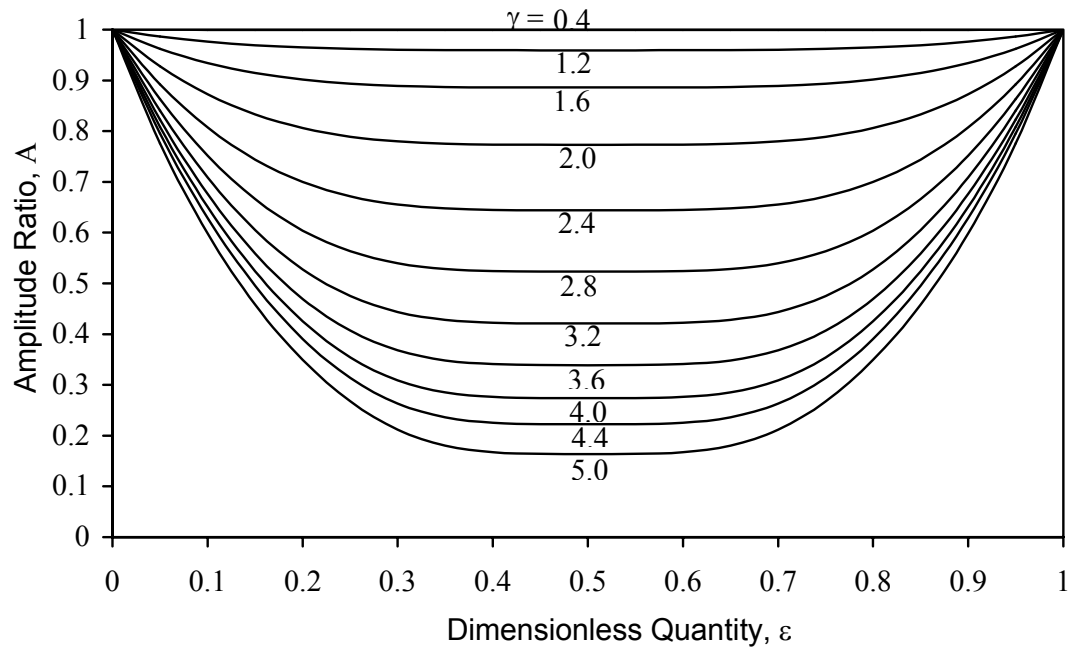


Figure 3.1 Theoretical plots for normalized amplitude ratio against the dimensionless quantity ε for various constant values of γ without lateral heat loss.

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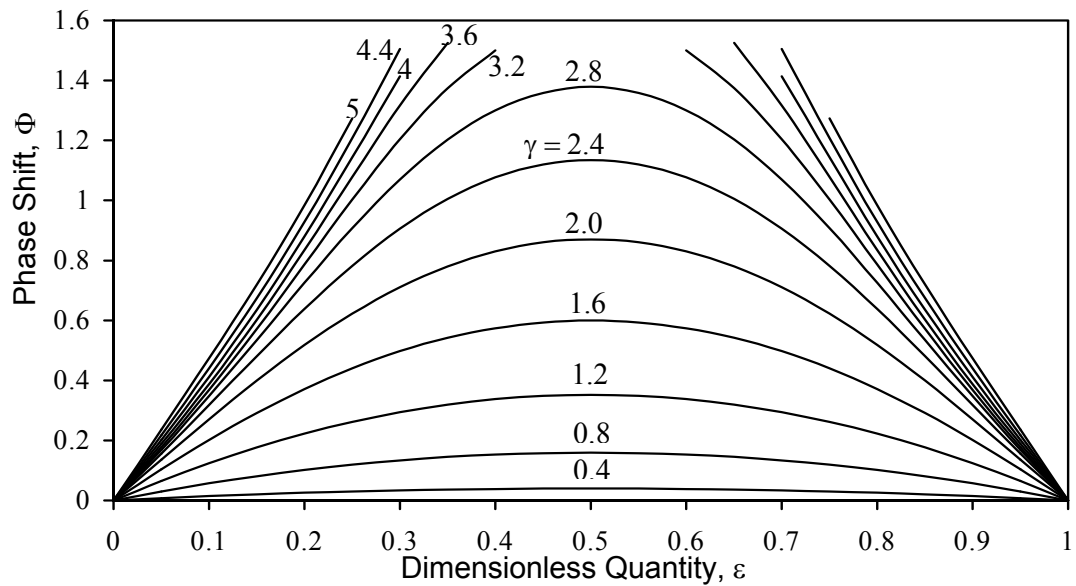


Figure 3.2 Theoretical plots for phase shift against the dimensionless quantity ε for various constant values of γ without lateral heat loss.

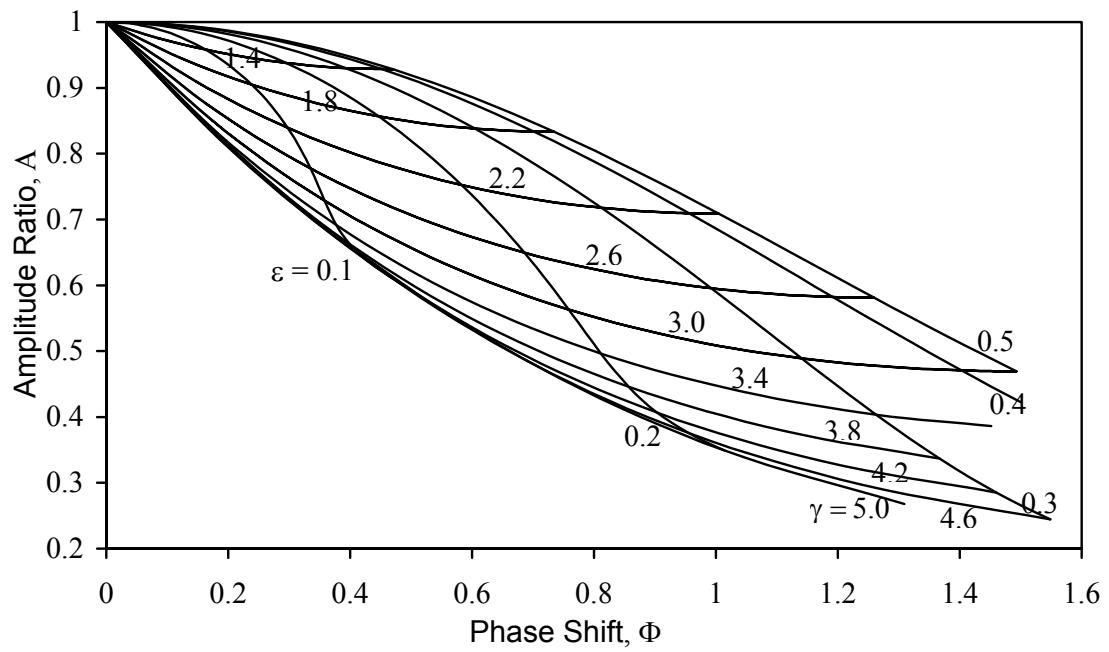


Figure 3.3 Theoretical plots for normalized amplitude ratio against phase shift for different values of ϵ and γ without lateral heat loss ($\beta = 0$).

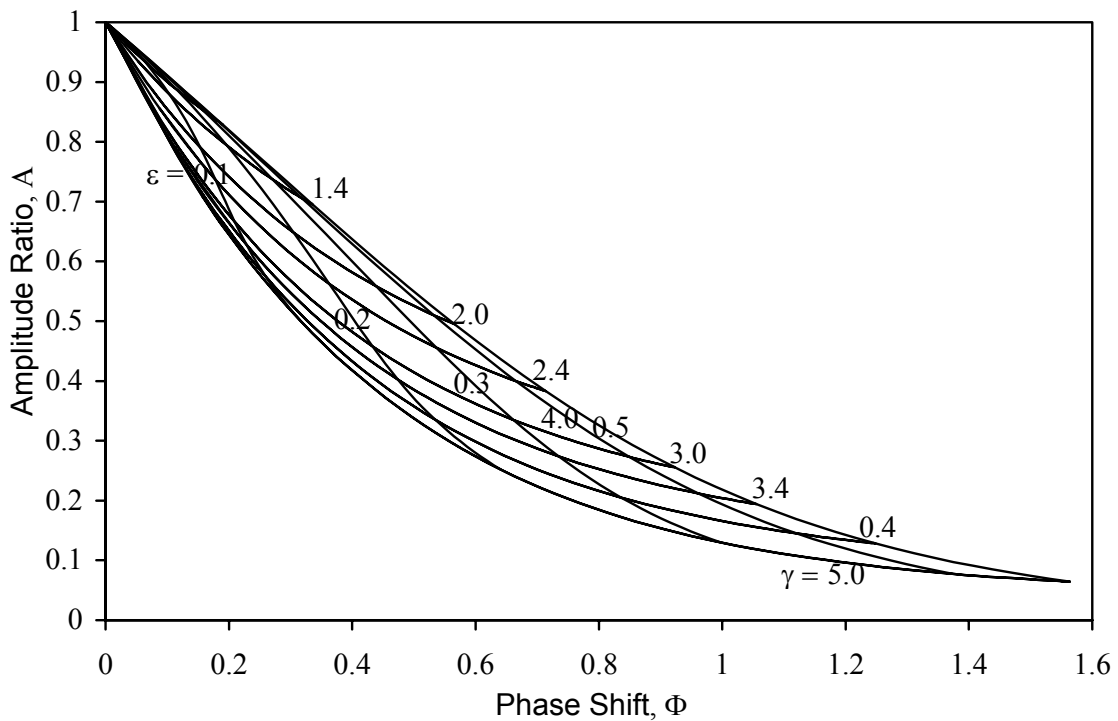


Figure 3.4 Theoretical plots for normalized amplitude ratio against the phase change for various constant values of ϵ and γ with lateral heat loss ($\beta = 0.01$).

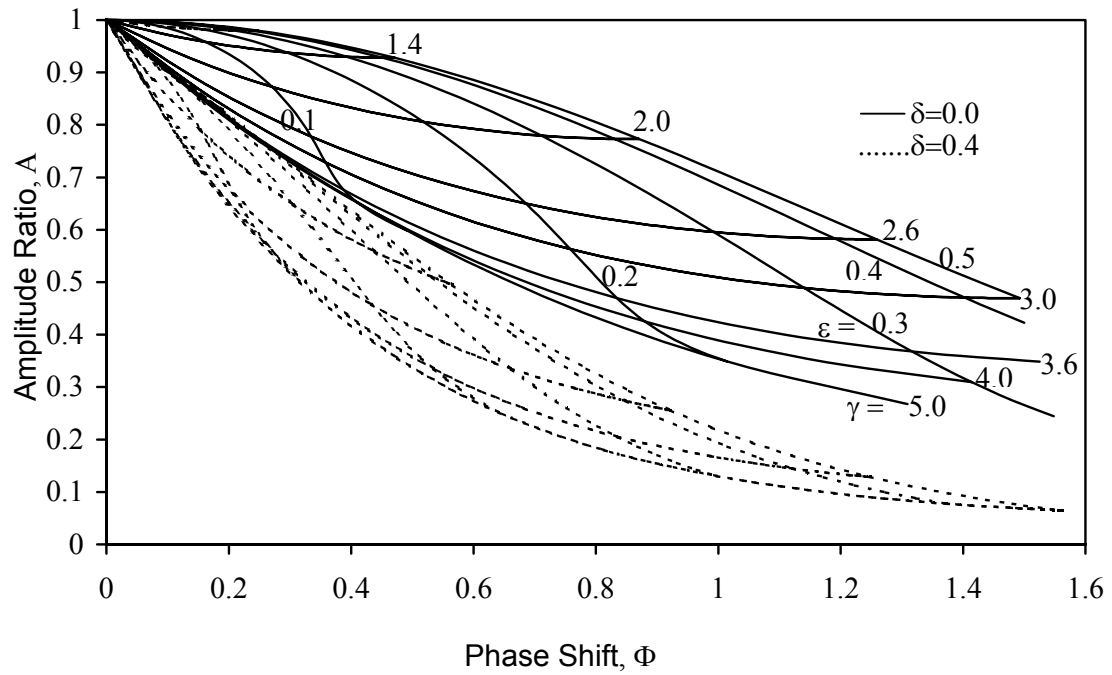


Figure 3.5 Theoretical plots for the normalized amplitude ratio against the phase change for various constant values of ϵ and γ with and without lateral heat losses.

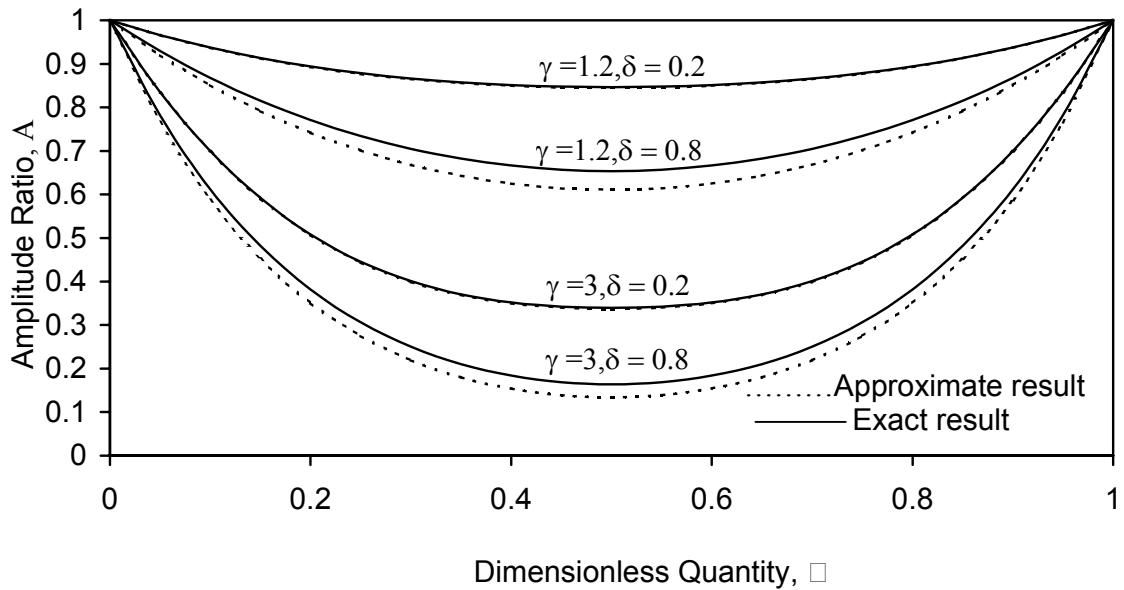


Figure 3.6 Comparisons between approximate solution and exact solution for amplitude ratio.

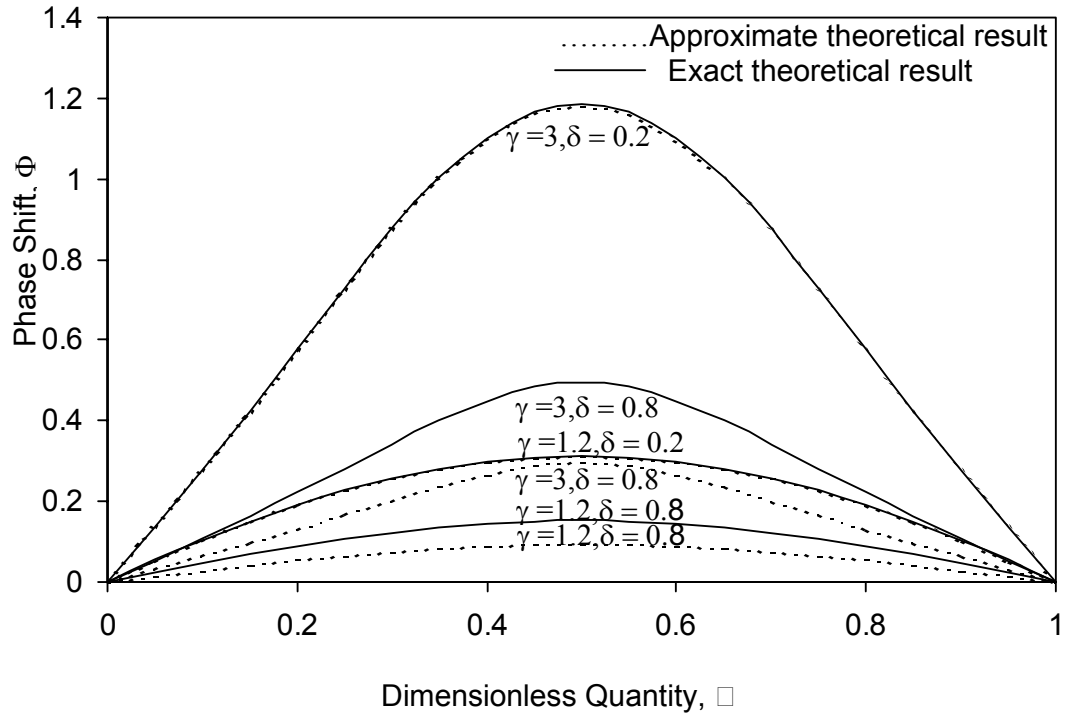


Figure 3.7 Comparisons between approximate solution and exact solution for phase shift.

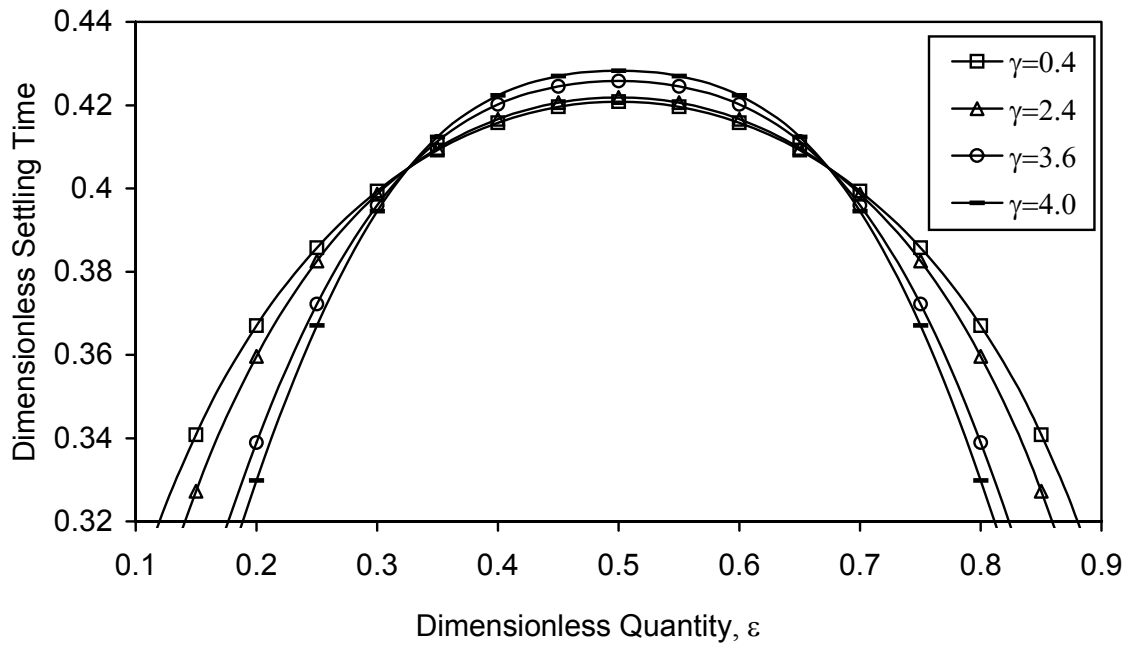


Figure 3.8 Theoretical plots for the dimensionless settling time against ε for various constant value of γ without lateral heat loss.

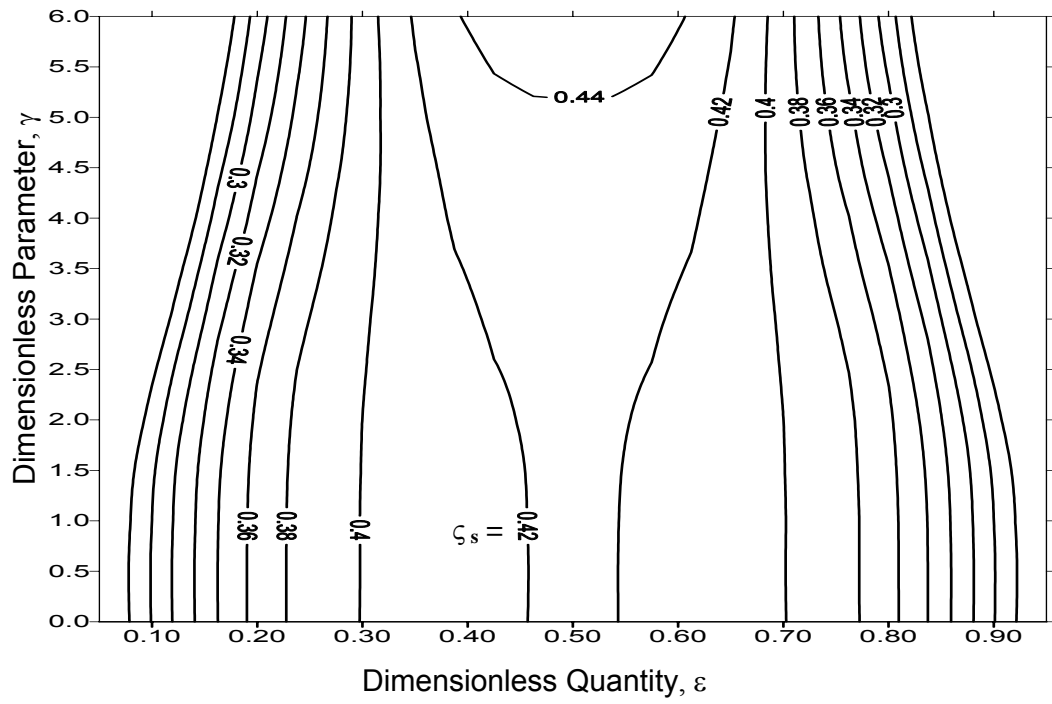


Figure 3.9 Contour plots of dimensionless settling time for different values of γ and δ without lateral heat loss.

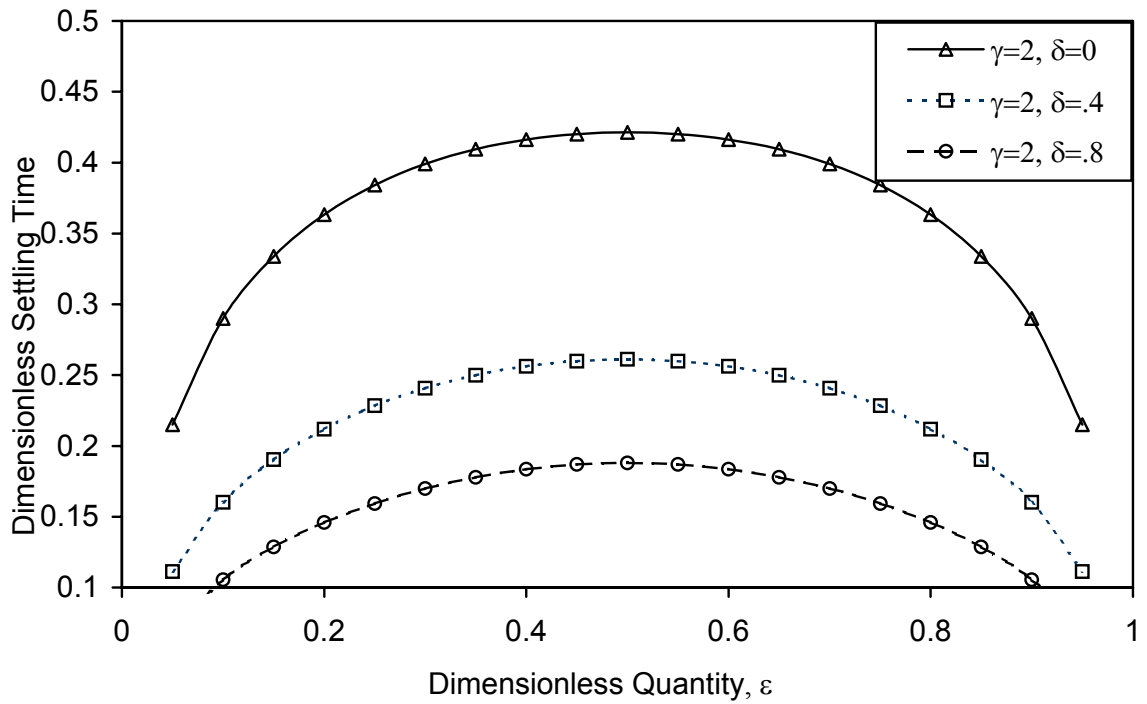


Figure 3.10 Theoretical plots for the dimensionless settling time against ϵ for various constant values of γ with and without lateral heat losses.

Chapter 4

Principles of Experiment for Measurement of Thermal Diffusivity

4.1 Introduction

Thermal diffusivity of a material is measured with a relatively simple, inexpensive and compact technique called temperature oscillation method (TOM). This technique combines the advantages of a steady state measurement with the potential to measure a property describing a non-steady state. The method is purely thermal and the electrical components of the apparatus are away from the test sample, which does not influence the experimental data. One of the major advantages of the temperature oscillation technique is that a high degree of accuracy of thermophysical properties are attainable at different temperatures ranging from liquid helium to high temperatures upon which oscillation is imposed. In this chapter, the theory of the technique to measure thermal diffusivity, theory of the associated Fourier analysis and its related uncertainty analysis are discussed.

The subsequent chapter describes the experimental procedure to yield the desired results based on the application of the basic principle developed in this chapter.

4.2 Mathematical Model for Measurement

A semi-infinite geometry of the test sample is chosen so as to have enough sample length to attenuate the input signal to a measurable quantity at the output point. The input was chosen very carefully to be a periodic oscillation since only for oscillatory inputs, the parabolic effects can persist after a comparatively long distance as opposed to instantaneous inputs such as step change used in some of the earlier experiments in the references. Due to the above consideration, for a semi-infinite sample geometry, a suitable predefined amplitude and frequency for the input boundary temperature variation has been chosen. A sketch of the coordinate system is shown in Fig. (4.1). To carry out the measurement of thermal diffusivity of liquid, the oscillatory inputs has got an additional advantages in that the two quantities of amplitude attenuation and phase shift can be measured. Thus two unknown quantities can be evaluated without the need of any iteration or curve fitting. This basic principle is used to measure thermal diffusivity. Additionally temperature recorded at a axial location of sample is used for modeling without any assumption of ideal input.

The mathematical model for parabolic one-dimensional conductive heat transfer in a semi-infinite medium is given by

$$\frac{\partial^2 T}{\partial x^2} = \frac{1}{\alpha} \frac{\partial T}{\partial t} \quad (4.1)$$

The length of the semi-infinite body in the x direction is so large that the temperature time variation at any depth x within the material will depend only on conditions imposed at x = 0. The boundary condition at surface of the body (at x = 0) is taken as sinusoidal surface temperature variations, namely

$$T = T_{m1} + T_0 \exp(i\omega t) \quad : x = 0, t > 0 \quad (4.1a)$$

Similarly, the boundary condition at the semi-infinite side can be depicted as,

$$T = T_{m1} \quad : x \rightarrow \infty, t > 0 \quad (4.1b)$$

T_{m1} is the mean temperature of oscillation, T_0 is the amplitude of temperature wave

and ω is the frequency of oscillation. It may be noted that at the mean temperature, the desired thermophysical properties are evaluated.

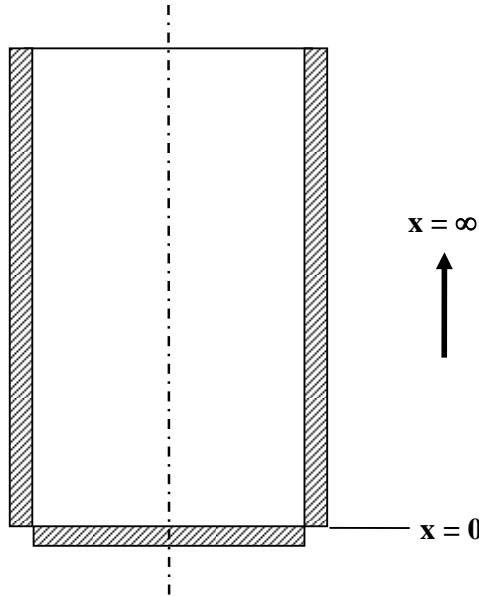


Figure 4.1 Sketch of the coordinate system.

Using temperature difference $\theta = T - T_{m1}$, the Eq. (4.1) and the associated boundary conditions can be reduced to the temperature difference form

$$\frac{\partial^2 \theta}{\partial x^2} = \frac{1}{\alpha} \frac{\partial \theta}{\partial t} \quad (4.2)$$

$$\theta = T_0 \exp(i\omega t) \quad : x = 0, t > 0 \quad (4.2a)$$

$$\theta = 0 \quad : x \rightarrow \infty, t > 0 \quad (4.2b)$$

4.2.1 Solution of Governing Equation for Semi-Infinite Medium

Taking the Laplace transform of the Eq. (4.2) along with its boundary conditions yields,

$$\frac{d^2 \bar{\theta}}{dx^2} = \frac{1}{\alpha} [s\bar{\theta} - \theta_{t=0}] \quad (4.3)$$

$$\bar{\theta} = \frac{T_0}{(s - i\omega)} \quad : x = 0, t > 0 \quad (4.3a)$$

$$\bar{\theta} = 0 \quad : x \rightarrow \infty, t > 0 \quad (4.3b)$$

The general solution of the Eq. (4.3) is

$$\bar{\theta} = C_9 \exp(m_2 x) + C_{10} \exp(-m_2 x) \quad (4.4)$$

$$\text{where } m_2 = \sqrt{(s/\alpha)} \quad (4.5)$$

The arbitrary constants C_9 and C_{10} which can be determined by using the boundary conditions.

Use of second boundary condition given by Eq. (4.3b), yields

$$C_9 = 0 \quad (4.6)$$

and applying first boundary condition given by Eq. (4.3a), yields

$$C_{10} = \frac{T_0}{(s - i\omega)} \quad (4.7)$$

Substituting the values of C_9 and C_{10} in Eq. (4.4) yields,

$$\bar{\theta} = \frac{T_0 \exp(-m_2 x)}{(s - i\omega)} \quad (4.8)$$

The Laplace inversion of this equation is required to be evaluated since this does not appear in the table of Laplace transform.

For the function $F(x)$ and its Laplace transform $F(s)$, are related by contour integral and residues as,

$$F(x) = \frac{1}{2\pi i} \int_{y-i\infty}^{y+i\infty} e^{st} F(s) ds = \Sigma \text{Residue of } e^{st} F(s) \quad (4.9)$$

$$\text{Therefore, } \theta = \frac{T_0}{2\pi i} \int_{y-i\infty}^{y+i\infty} \frac{\exp(st) \exp(-x\sqrt{s/\alpha}) ds}{(s - i\omega)} \quad (4.10)$$

This integrand has a branch point at $s = 0$ and a simple pole at $(s = i\omega)$

The residue at the pole, $s = i\omega$ is

$$R_{es}(s=i\omega) = T_0 \exp(i\omega t) \exp(-x\sqrt{i\omega/\alpha}) \quad (4.11)$$

To convert the Eq. (4.10) as a single valued function, the integrand given in Eq. (4.10) is to be modified as,

$$\theta = \frac{T_0}{2\pi i} \oint_{ABFEDCA} \frac{\exp(st)}{(s - i\omega)} \exp(-x\sqrt{s/\alpha}) ds \quad (4.12)$$

where the integration path, ABFEDCA as shown in Fig. (4.2) avoids the branch point at $s = 0$.

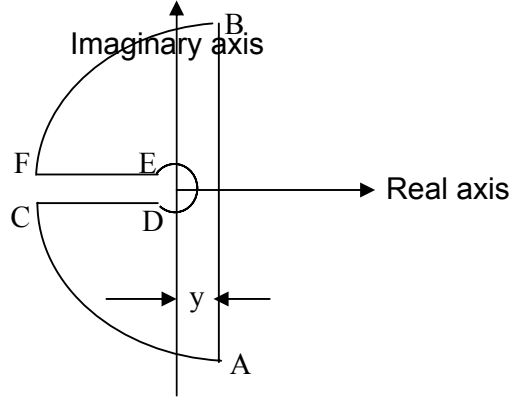


Figure 4.2 Contour integral for a branch point at $s=0$.

The integral around the contour ABFEDCA (Fig. 4.2) is the contour integral for Laplace inversion in the limit as the radius R of the large circle tends to infinite, and that of the small circle tends to zero. As $R \rightarrow \infty$, the integral over the arcs BF and CA tends to zero. Also the integral over the small circle about the origin tends to zero. As $R \rightarrow \infty$, the integral over AB becomes the Bromwich integral used for the inversion.

The integral along EF and CD, $s = \rho \exp(i\pi)$ and $s = \rho \exp(-i\pi)$ are substituted respectively. Here the argument (angle) of s is π on EF and $-\pi$ on CD and ρ is any real value varies from $-\infty$ to 0 and 0 to $-\infty$ respectively.

Along FE, $s = \rho e^{i\pi}$, thus,

$$\int_{FE} \frac{\exp(-x\sqrt{s/\alpha}) \exp(st)}{(s-i\omega)} ds = \int_{\infty}^0 \frac{\exp(-ix\sqrt{\rho/\alpha}) \exp(-\rho t)(-d\rho)}{(-\rho-i\omega)} \quad (4.13)$$

Along CD, $s = \rho e^{-i\pi}$, thus,

$$\int_{DC} \frac{\exp(-x\sqrt{s/\alpha}) \exp(st)}{(s-i\omega)} ds = \int_0^{\infty} \frac{\exp(ix\sqrt{\rho/\alpha}) \exp(-\rho t)(-d\rho)}{(-\rho-i\omega)} \quad (4.14)$$

Sum of integrand of FE and CD is

$$\int_{FE} + \int_{DC} = 2i \int_0^{\infty} \frac{\rho-i\omega}{\rho^2 + \omega^2} \exp(-\rho t) \sin(x\sqrt{\rho/\alpha}) d\rho \quad (4.15)$$

Thus the Bromwich integral can be written as,

$$\frac{T_0}{2\pi i} \int_{\gamma-i\infty}^{\gamma+i\infty} \frac{\exp(st) \exp(-x\sqrt{s/\alpha}) ds}{(s-i\omega)} = T_0 \exp i(\omega t - x\sqrt{\omega/2\alpha}) \exp(-x\sqrt{\omega/2\alpha}) - \frac{T_0}{2\pi i} 2i \int_0^\infty \frac{\rho-i\omega}{\rho^2 + \omega^2} \exp(-\rho t) \sin(x\sqrt{\rho/\alpha}) d\rho \quad (4.16)$$

Finally, the solution of Eq. (4.2) is

$$\theta = T_0 \exp i(\omega t - x\sqrt{\omega/2\alpha}) \exp(-x\sqrt{\omega/2\alpha}) - \frac{T_0}{2\pi i} 2i \int_0^\infty \frac{\rho-i\omega}{\rho^2 + \omega^2} \exp(-\rho t) \sin(x\sqrt{\rho/\alpha}) d\rho \quad (4.17)$$

The Eq. (4.17) is the response for a signal of $T_0 \exp(i\omega t)$.

For a unit signal input, the response is,

$$I(\theta) = \left\{ \exp(-ix\sqrt{\omega/2\alpha}) \exp(-x\sqrt{\omega/2\alpha}) \right\} \left[\frac{\exp(-i\omega t)}{\pi} \int_0^\infty \frac{\rho-i\omega}{\rho^2 + \omega^2} \exp(-\rho t) \sin(x\sqrt{\rho/\alpha}) d\rho \right] \quad (4.18)$$

The first term of the Eq. (4.18) is the steady periodic solution and the second term is the transient part. For large value of time all transient disturbances fade away and only the remaining steady periodic solutions are required to measure the amplitude ratio and phase change. The steady periodic response for unit signal input is

$$I(\theta_s) = \exp(-ix\sqrt{\omega/2\alpha}) \exp(-x\sqrt{\omega/2\alpha}) \quad (4.19)$$

By separation of real and imaginary part, the real amplitude response and phase shift can be expressed as,

$$A = \exp(-x\sqrt{\omega/2\alpha}) \quad (4.20)$$

$$\text{and} \quad \Phi = x\sqrt{\omega/2\alpha} \quad (4.21)$$

Measurement of the amplitude ratio or the phase difference allows the thermal diffusivity to be determined from Eq. (4.20) or Eq. (4.21), respectively.

4.2.2 Settling Time

In the transient technique, the time to achieve the pseudo-steady state is an important factor known as settling time. Under the steady state condition, the

amplitude ratio or phase change is used to determine the thermophysical properties. It may be noted that collecting data earlier to this settling time may include erroneous values. For a reasonable approximation in practice, the steady state condition is obtained when time required for the temperature of the transient part to achieve 2% of the steady state temperature.

The response for unit signal input, given by Eq. (4.18) can be simplified by using the following variables:

$$\rho = \omega u^2, \quad \omega t = \omega^*, \quad x\sqrt{\omega/2\alpha} = \beta^* \quad (4.22)$$

$$\text{where,} \quad d\rho = 2\omega u \, du \quad (4.23)$$

Thus the steady part is,

$$I(\theta_s) = \exp(-\beta^*) \exp(i\beta^*) \quad (4.24)$$

and the transient part is,

$$I(\theta_T) = \exp(-i\omega^*) \frac{1}{\pi} \int_0^\infty \frac{i - u^2}{1 + u^4} \exp(-u^2 \omega^*) \sin(\beta^* \sqrt{2} u) 2u \, du \quad (4.25)$$

Separating the real and imaginary parts, the real and imaginary integrals can be expressed as,

$$\text{Re}[I(\theta_T)] = \frac{2}{\pi} \int_0^\infty \frac{\sin \omega^* + u^2 \cos \omega^*}{1 + u^4} \exp(-u^2 \omega^*) \sin(\beta^* \sqrt{2} u) u \, du \quad (4.26)$$

$$\text{Im}[I(\theta_T)] = \frac{2}{\pi} \int_0^\infty \frac{\cos \omega^* + u^2 \sin \omega^*}{1 + u^4} \exp(-u^2 \omega^*) \sin(\beta^* \sqrt{2} u) u \, du \quad (4.27)$$

Thus the magnitude of the temperature amplitude in the transient Eq. (4.25) is given by $(\theta_T)_R = \sqrt{\{[\text{Re}[I(\theta_T)]]\}^2 + \{[\text{Im}[I(\theta_T)]]\}^2}$.

As per the condition given earlier, the equation for the settling time can be expressed as: $0.02 \exp(-\beta^*) - \sqrt{\{[\text{Re}[I(\theta_T)]]\}^2 + \{[\text{Im}[I(\theta_T)]]\}^2} = 0$ (4.28)

The solution of 't' from the Eq. (4.28) determines the settling time. For a low thermal conductivity material the settling time is an appreciable quantity. A more detailed analysis for a finite system has been shown in Chapter 3. In the present situation, this value will be order of seconds, which will be of no use to rigorously calculate from Eq. (4.28). Also this supposition is evident from the experiment. However, these derivations are presented to show the mathematical completeness.

4.3 Fourier Analysis

The temperature oscillation, in this experiment is obtained by TEC (Thermo Electric Cooler), which is a Peltier element. By applying a square pulse or sinusoidal voltage to the Peltier element whose one side is kept at a constant temperature bath, the other side of the Peltier element generates temperature wave similar to the applied voltage. Additionally, if the applied voltage is modulated over a D.C. component, the temperature wave of oscillating part modulated over a mean temperature corresponding to D.C. component of applied oscillating voltage is obtained. Due to distortion, the temperature generated may not be of same shape as the applied voltage. Also due to the change in wave shape, the shape of temperature wave at the input to a sample may distort at the output from the sample.

To overcome the distortion of the temperature wave Fourier analysis is applied. The Fourier analysis [151,152] converts the distorted signal to sinusoidal signals where the fundamental frequency is relevant to the analysis.

In general, a periodic function of an oscillating temperature signal can be expressed in Fourier series as,

$$T = \frac{1}{2}a_0 + \sum_{n=1}^{n=\infty} a_n \cos(n\omega t) + \sum_{n=1}^{n=\infty} b_n \sin(n\omega t) \quad (4.29)$$

where a_0 , a_n and b_n are the Fourier coefficients defined as,

$$a_n = \frac{2}{t_p} \int_{-t_p/2}^{t_p/2} T(t) \cos(n\omega t) dt, \quad n=0,1,2,3,\dots \quad (4.30)$$

$$b_n = \frac{2}{t_p} \int_{-t_p/2}^{t_p/2} T(t) \sin(n\omega t) dt, \quad n=1,2,3,\dots \quad (4.31)$$

The Fourier series can also be written as the sum of cosine terms only. This can be obtained by substituting $a_n = A_n \cos \Phi_n$ and $b_n = A_n \sin \Phi_n$ in Eq. (4.29), thus

$$T = \frac{a_0}{2} + \sum_{n=1}^{n=\infty} A_n \cos(n\omega t - \Phi_n) \quad (4.32)$$

where $A_n = \sqrt{(a_n^2 + b_n^2)}$ (4.33)

$$\Phi_n = \arctan(b_n/a_n) \quad (4.34)$$

Using Euler identity,

$$e^{in\omega t} = \cos(n\omega t) + i \sin(n\omega t) \quad (4.35)$$

Equation (4.29) can be rewritten as,

$$T = \frac{1}{2}a_0 + \sum_{n=1}^{n=\infty} \left(\frac{a_n - ib_n}{2} e^{in\omega t} + \frac{a_n + ib_n}{2} e^{-in\omega t} \right) \quad (4.36)$$

or
$$T = \sum_{n=-\infty}^{n=\infty} C_n e^{in\omega t} \quad (4.37)$$

where
$$C_n = \frac{a_n - ib_n}{2} \text{ and } C_{-n} = \frac{a_n + ib_n}{2} \quad (4.38)$$

Hence
$$a_n = C_n + C_{-n} \quad (4.39)$$

and
$$b_n = i(C_n - C_{-n}) \quad (4.40)$$

For real function (T), C_n and C_{-n} are the complex conjugate, where a_n and b_n are real quantities.

For an arbitrary periodic temperature signal, T, its FFT analysis gives the coefficients C_n ($n=0,1,2,\dots$) where C_{-n} can be estimated by taking the complex conjugate of C_n . Thus the first value of the FFT spectrum (C_0) gives the D.C. component, which is equal to the mean temperature. The amplitude and phase can be calculated from Eqs. (4.33) and (4.34) as,

$$A_n = \sqrt{(\text{Re}[C_n])^2 + (\text{Im}[C_n])^2} \quad (4.41)$$

and
$$\Phi_n = \arctan \frac{\text{Im}[C_n]}{\text{Re}[C_n]} \quad (4.42)$$

In the measurement of amplitude and phase the fundamental frequency is considered. In FFT [151, 152] technique these values are obtained from Equations (4.41) and (4.42) for the fundamental frequency with $n=1$.

4.4 Theory of Error Analysis

Measurement Error:

It is a well-accepted principle in engineering that all measurements have errors (δ_k). These errors are the differences between the measurements and the true value (see Fig. 4.3). Furthermore, the total error is usually expressed in terms

of two components: a fixed (bias) error (B), and a random (precision) error (e_k) such that

$$\delta_k = B + e_k \quad (4.43)$$

Precision Index:

The precision error is determined by taking N repeated measurements from the parameter population, the characteristics of which can be approximated by the precision index (S_X) defined by the familiar expression,

$$S_X = \sqrt{\frac{\sum_{k=1}^{k=N} (X_k - \bar{X})^2}{(N-1)}} \quad (4.44)$$

where \bar{X} is the average value of X .

The precision index of the average of a set of N separate measurement readings is determined as,

$$S_{\bar{X}} = \frac{S_X}{\sqrt{N}} = \sqrt{\frac{\sum_{k=1}^{k=N} (X_k - \bar{X})^2}{N(N-1)}} \quad (4.45)$$

The associated number of degrees of freedom from t -distribution is equal to $N-1$.

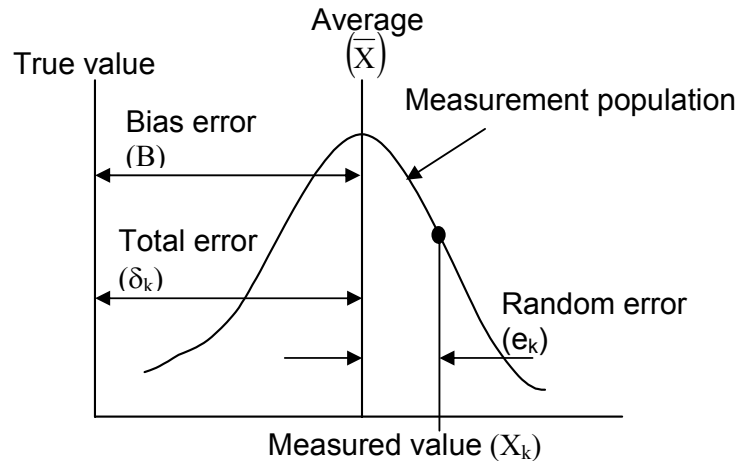


Figure 4.3 Measurement errors.

Bias error:

The bias error is the systematic error, which is considered to remain constant during a given test. Thus, in repeated measurements of a given set, each

measurement has the same bias. There is no statistical equation, as (4.44) or (4.45), to define the bias limit, B . Instead, it must be estimated and it is not an easy matter since the true value is not known. Calibration helps, as does a comparison of measurements in independent methods, but in general the estimate of bias must be based on judgments.

Combining errors due to elemental error sources:

Errors arise from many sources. These are divided arbitrarily into three categories [139]: calibration errors, data acquisition errors and data reduction errors. In each of these sources of error there will be bias and precision components.

To obtain the precision of a given parameter, J (like temperature, pressure, or flow rate), the root sum square (RSS) method is used to combine the precision indices from the M sources of error. Thus

$$S_J = \left[(S_J)_1^2 + (S_J)_2^2 + \dots + (S_J)_M^2 \right]^{1/2}. \quad (4.46)$$

$$S_J = (S_J)_{\bar{X}_i} = \frac{(S_J)_{X_i}}{\sqrt{N}} \quad (4.47)$$

$$\text{and} \quad (S_J)_{X_i} = \sqrt{\frac{\sum_{k=1}^{k=N} (X_k - \bar{X})^2}{(N-1)}} \quad (4.48)$$

The number of degrees of freedom associated with S_J is calculated using the Welch-Satterthwaite formula as,

$$v_J = \frac{\left[\sum_{k=1}^M (S_J)_k^2 \right]^2}{\sum_{k=1}^M \left[(S_J)_k^4 / (v_J)_k \right]} \quad (4.49)$$

$$\text{where,} \quad (v_J)_k = (N_j)_k - 1 \quad (4.50)$$

Similarly, the bias of a given parameter is given by

$$B_J = \left[(B_J)_1^2 + (B_J)_2^2 + \dots + (B_J)_M^2 \right]^{1/2} \quad (4.51)$$

Uncertainty of a result:

Once the precision index and associated degrees of freedom have been determined for each of the variables X_i in the data reduction equation, the precision index of the result, S_r , is found from the given functional uncertainty analysis expression

$$S_r = \left[\left(\frac{\partial r}{\partial X_1} S_1 \right)^2 + \left(\frac{\partial r}{\partial X_2} S_2 \right)^2 + \dots + \left(\frac{\partial r}{\partial X_J} S_J \right)^2 \right]^{1/2} \quad (4.52)$$

The number of degrees of freedom, v_r , associated with S_r is then determined from the Welch-Satterthwaite formula that may be written as,

$$v_r = \frac{\left[\sum_{i=1}^J (\theta_i S_i)^2 \right]^2}{\sum_{i=1}^J [(\theta_i S_i)^4 / (v_i)]} = \frac{S_r^4}{\sum_{i=1}^J [(\theta_i S_i)^4 / (v_i)]} \quad (4.53)$$

or in the alternate form

$$v_r = \frac{\left\{ \sum_{i=1}^J [\theta_i' (S_i / X_i)]^2 \right\}^2}{\sum_{i=1}^J [(\theta_i' S_i / X_i)^4 / v_i]} = \frac{(S_r / r)^4}{\sum_{i=1}^J [(\theta_i' S_i / X_i)^4 / v_i]} \quad (4.54)$$

where, the $\theta_i S$ is defined as

$$\theta_i = \partial r / \partial X_i \quad (4.55)$$

and the θ_i' is defined as

$$\theta_i' = \frac{\partial r / r}{\partial X_i / X_i} = \left(\frac{X_i}{r} \right) \theta_i \quad (4.56)$$

The appropriate value of t_d is then found from the table for the t-distribution with degrees of freedom, v_r . Similar to Eq. (4.52), the bias limit of a result is given by

$$B_r = \left[\left(\frac{\partial r}{\partial X_1} B_1 \right)^2 + \left(\frac{\partial r}{\partial X_2} B_2 \right)^2 + \dots + \left(\frac{\partial r}{\partial X_J} B_J \right)^2 \right]^{1/2} \quad (4.57)$$

The uncertainty of a result for the two models are given as

$$U_{r, \text{ADD}} = B_r + t_d S_r, \text{ @ 99\% coverage} \quad (4.58)$$

$$\text{and } U_{r, \text{RSS}} = \left[B_r^2 + (t_d S_r)^2 \right]^{0.5}, \text{ @ 95\% coverage} \quad (4.59)$$

The student t_d value is a function of the degrees of freedom used in calculating the total contribution due to S_r . For large sample size, (i.e., $N > 30$), t_d is set equal to 2, otherwise, for small sample sizes of $N \leq 30$, t_d is calculated from the Welch-Satterthwaite formula.

Uncertainty in measurement of thermal diffusivity:

The functional relation for the measurement of thermal diffusivity as stated by Eq. (4.20) can be rewritten as,

$$\alpha = \frac{x^2 \pi}{t_p (\log_e A)^2} \quad (4.60)$$

The measurement errors associated with this equation are

$$\delta \alpha = \left[\left(\frac{\partial \alpha}{\partial x} \right)^2 \delta x^2 + \left(\frac{\partial \alpha}{\partial t_p} \right)^2 \delta t_p^2 + \left(\frac{\partial \alpha}{\partial A} \right)^2 \delta A^2 \right]^{0.5} \quad (4.61)$$

$$\text{where } \frac{\partial \alpha}{\partial x} = \frac{2x \pi}{t_p (\log_e A)^2}, \quad \frac{\partial \alpha}{\partial t_p} = -\frac{x^2 \pi}{t_p^2 (\log_e A)^2},$$

$$\text{and } \frac{\partial \alpha}{\partial A} = -\frac{2x^2 \pi}{A t_p (\log_e A)^3}. \quad (4.62)$$

Dividing Eq. (4.61) by α and substituting the value of partial derivative leads to

$$\frac{\delta \alpha}{\alpha} = \left[\left(\frac{2\delta x}{x} \right)^2 + \left(\frac{\delta t_p}{t_p} \right)^2 + \left(\frac{2\delta A}{A \log_e A} \right)^2 \right]^{0.5} \quad (4.63)$$

where $\delta x/x$, $\delta t_p/t_p$ and $\delta A/A$ are the fractional errors in the distance between two thermocouples, periodic time measurement and amplitude ratio respectively.

The amplitude ratio is not directly measured but it is obtained from temperature data recorded by thermocouple. Hence, the bias error propagated in measurement of amplitude ratio can be obtained as,

$$\frac{\delta A}{A} = \sqrt{\left(\frac{\delta T_{\text{out}}}{T_{\text{out}}}\right)^2 + \left(\frac{\delta T_{\text{in}}}{T_{\text{in}}}\right)^2} \quad (4.64)$$

where $\delta T_{\text{out}}/T_{\text{out}}$, $\delta T_{\text{in}}/T_{\text{in}}$ are the fractional errors in output and input temperature amplitudes response.

In the present experimental procedure, it is assumed that there is a single unknown source ($M=1$) of error for the measurement of precision and bias errors. The number of parameters are distance, time period and amplitude ratio.

Measurement of precision error:

From the Eq. (4.46) with $M=1$, the precision error contributions are S_1 , S_2 and S_3 for the above mentioned three parameters. The generalized Eq. (4.52) for the measurement of thermal diffusivity is expressed by Eq. (4.63). S_1 , S_2 and S_3 are the contribution of precision errors for distance, time period and amplitude ratio respectively as stated by Eq. (4.63). For the same sample holder and same wave generator, the contributions of precision errors for the first two parameters can be assumed to be zero. Thus the precision error is controlled by the parameter S_3 , which is the amplitude ratio. Hence the degrees of freedom for the single parameter S_3 are $N - 1$ and this value is used to calculate t_d from the t-distribution table.

Measurement of bias error:

Similar to precision error for $M=1$, the contributions of bias errors are B_1 , B_2 and B_3 for the three parameters: distance, time period and amplitude ratio respectively. For the measurement of bias error in thermal diffusivity, Equations (4.63) and (4.64) are considered by taking the measurement errors in distance, time period and thermocouple.

For the measurement of total uncertainty Eq. (4.59) is normally accepted. Based on the above theory, the step by step procedure has been illustrated as a specimen calculation in the subsequent chapter.

4.5 Discussions

The theoretical model for experimental measurement of thermal diffusivity has been presented. The boundary condition of the model is implemented experimentally as sinusoidal temperature oscillation at one end and the other end behaves like a semi-infinite medium. In semi-infinite medium, measurement of thermal diffusivity is possible either from amplitude attenuation or phase shifting of the propagating thermal wave imposed at one side of the sample.

A theoretical expression has been derived to estimate the settling time for any specimen. The settling time is an important time parameter, which indicates elapsed time after which the steady state data is to be collected. However considering the present nature of specimen which has the moderate thermal conductivity, this settling time will be of order of second and hence no calculation has been shown in this regard.

The Fourier analysis of periodic temperature wave has been adopted to obtain its amplitude and phase for the fundamental frequency. This can be carried out by Fast Fourier Transform of the data recorded by thermocouples at input and output. From the characteristic of thermal wave propagation, the thermal diffusivity of the specimen can be calculated from the attenuation of amplitude or from the phase lag of the thermal wave.

A measurement result without accompanying statement of uncertainty is incomplete. However no measurement should be considered exactly; it can only provide an estimate of the measurand. Thus, a systematic procedure to measure uncertainty in error propagation in the experimental result has been presented. Based on this procedure a specimen calculation has been provided in the next chapter.

Chapter 5

Experimental Studies on the Measurement of Thermal Diffusivity

5.1 Introduction

Thermophysical properties, such as thermal conductivity and thermal diffusivity, are very important liquid properties. Accurate values of these properties are critical for practical engineering design as well as theoretical studies and analysis, especially in the fields of heat transfer and thermal processing.

Due to the unique characteristics of liquids, measurement of the thermal conductivity and thermal diffusivity is more challenging for liquids than for solids. Liquids do not maintain any fixed shape and can be easily changed compositionally, which alters their properties. Also, since liquids cannot sustain a shear stress, convection can occur in the presence of temperature gradients, which is one of the major error sources for many conventional techniques that measure liquid thermal conductivity and thermal diffusivity.

In this chapter, measurement of thermal diffusivity of liquid is presented based on the principle outlined in the previous chapter. Considering the intricacy in the measurement of thermal diffusivity of liquids, a considerable attention has been given in designing the sample holder along with its accessories and measuring

instruments. Measurement of thermal diffusivity of four liquids: ethylene glycol, ethanol, glycerol and water have been undertaken and the results have been compared with the published data. This chapter comprises description of apparatus, experimental data analysis and error measurement in more detail.

5.2 Experimental Setup

The experimental setup comprises of the fabricated test cell, constant temperature bath unit, signal generator with amplifier, and the instruments for the measurement system as shown in Figs. (5.1) to (5.2). For the semi-infinite body, the amplitude of temperature oscillation diminishes according to Eq. (4.20). Thus one thermal wavelength may be defined as,

$$\ell = \sqrt{4\pi\alpha t_p} \quad (5.1)$$

At a distance of one thermal wavelength, the amplitude is reduced by a factor of $\exp(-2\pi) = 0.0019$. It shows that the waves are very strongly attenuated. This implies that the solution of the semi-infinite specimen can be used for a specimen whose length is greater than one or two wavelengths. Thus, the space length of the sample has been determined from Eq. (5.1). The measurement system consists of specially fabricated test cell, thermostatic bath, power supply system and signal generator, and data acquisition system. These components are described below.

5.2.1 Test Cell

The schematic diagram of test cell is shown in Fig. (5.3) and its photograph in experimental setup shown in plates (5.4) and (5.5). In the Fig. (5.3) the fabricated test cell consists of a cylindrical well bore in a block (1) of POM (polyoxymethylene material), which can be machined and drilled accurately. The diameter of the bore is 26 mm, outer diameter 70 mm and the length 52 mm. It contains test liquid, which acts as a semi-infinite medium. The cavity is closed on both the sides by the removable discs (2) of copper material of dimension 26 mm diameter and 12 mm axial length. Thus, the space for the sample has a dimension of 26 mm diameter and 28 mm length. For filling the sample liquid, a small hole is provided on the body of the test cell. The copper discs act as heating and cooling surface respectively. Between the disc (2) and the water-cooling chamber (4), a Peltier element (3)

(melcore type, CP-07) square 40 mm x 40 mm is sandwiched which generates the required temperature oscillation with the help of a function generator attached to the D.C. supply. The Peltier elements are thermoelectric modules, which are solid-state devices (no moving parts) that convert electrical signal into a temperature gradient. The electrical signal supplied through the function generator is consists of oscillating voltage of the required frequency modulated over a D.C. voltage. The cooling chambers located at the outer surface of the Peltier element are maintained at a constant temperature by circulating water from the constant temperature bath. Thus the Peltier element whose one side is kept at a constant temperature generates a regulated oscillating temperature over a mean temperature. For a semi-infinite system, the Peltier element at the far end of the sample is excited with a D. C. signal to maintain a constant mean temperature along the sample length.

At the interface of the sample material and the copper disc, a thermocouple (5) of 0.2 mm wire diameter is affixed. This represents the response of input signal. For the output signal in the thermocouple probe of 0.5 mm diameter is inserted at a distance of 5 mm in the sample holder. The third thermocouple is placed at a similar location as that of the first one to ensure semi-infinite assumption of the sample and to correct the mean temperature. All the thermocouples are K-type and these are located along the axis of the test cell. The signals of the thermocouples are amplified and filtered before connecting to data acquisition system, which in turn connected to a PC for recording the data.

5.2.2 Thermostatic Bath

A constant temperature bath is shown in Fig. (5.6). This unit comprises a cooling system, heating system, temperature controller and water circulating pump. It is an automatic control device system, which maintains constant temperature between 0.0°C to 50°C with an accuracy of 0.1°C . The function of the temperature bath is to maintain a constant temperature at one side face of the Peltier element by water circulation in the cooling chamber. The constant temperature of the bath is used to maintain a constant mean temperature at the other surface of the Peltier element. However any further control of the mean temperature is done by controlling the D.C. component of the modulated signal feed to the Peltier element.

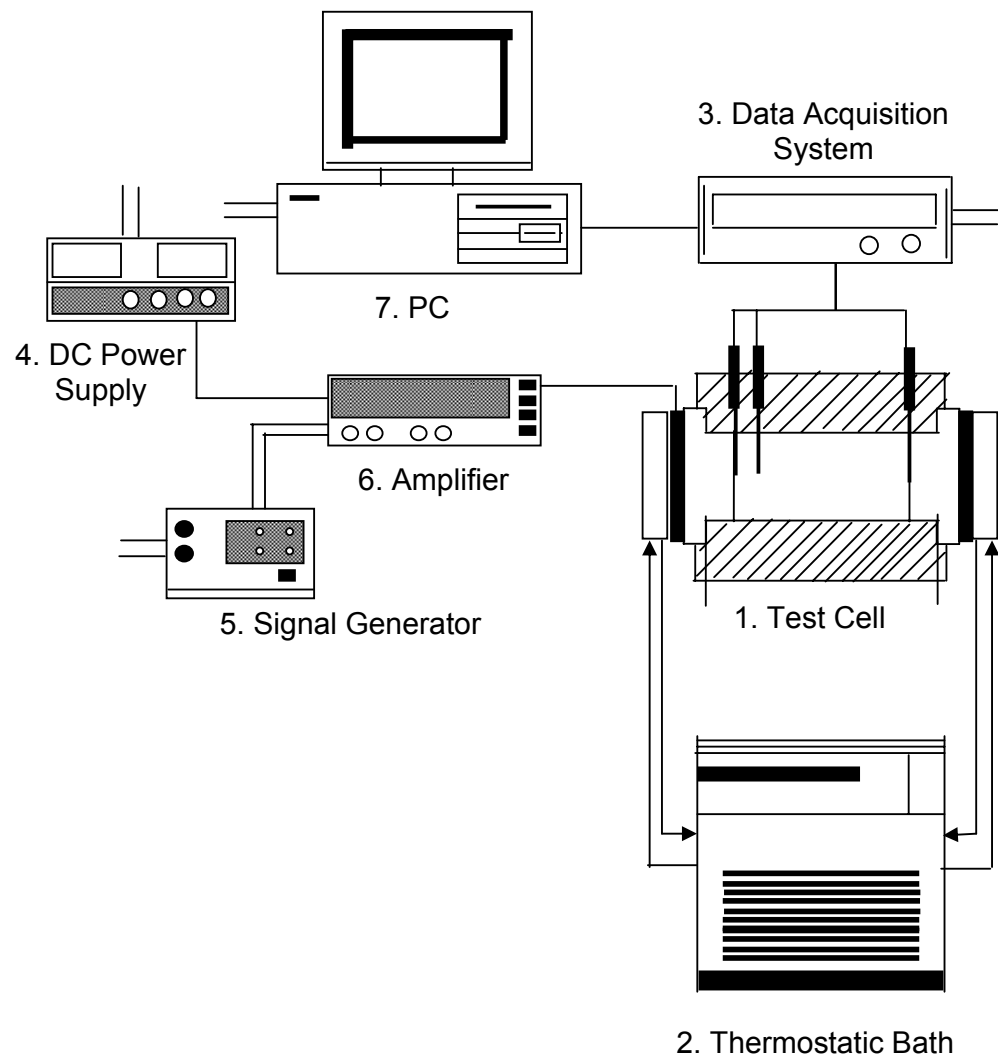


Figure 5.1 Schematic diagram of the experimental setup for the measurement of thermal diffusivity of liquids.

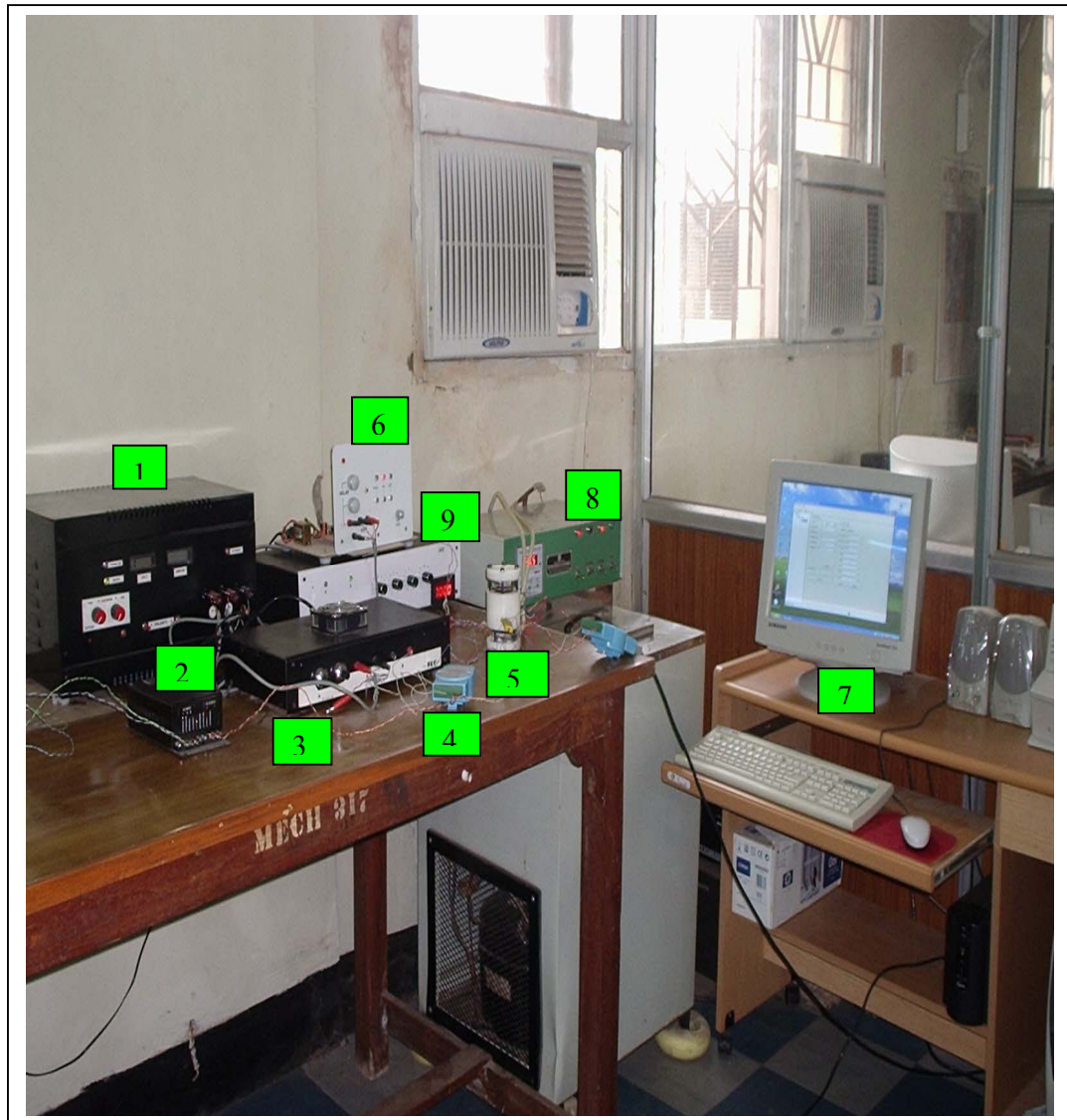


Figure 5.2 Photograph of the experimental setup: (1) DC power supply, (2) power supply for ADAM Module, (3) amplifier, (4) ADAM 4018, (5) test cell, (6) signal generator, (7) PC, (8) thermostatic bath, (9) phase shifter.

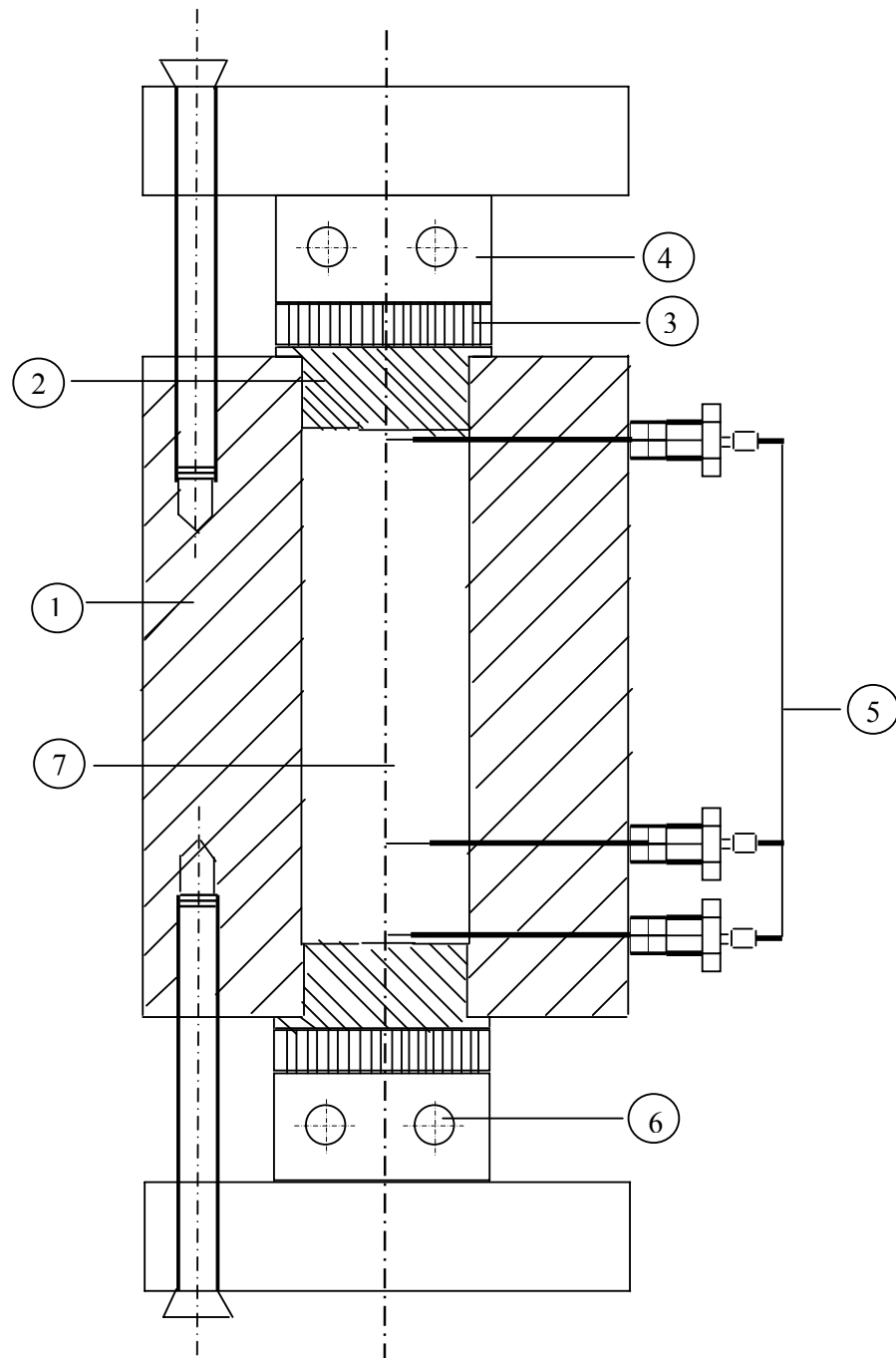


Figure 5.3 Fabricated Test Cell: (1) specimen holder, (2) removable disk, (3) Peltier element, (4) heat sink, (5) thermocouple, (6) water circulating valve (7) specimen.

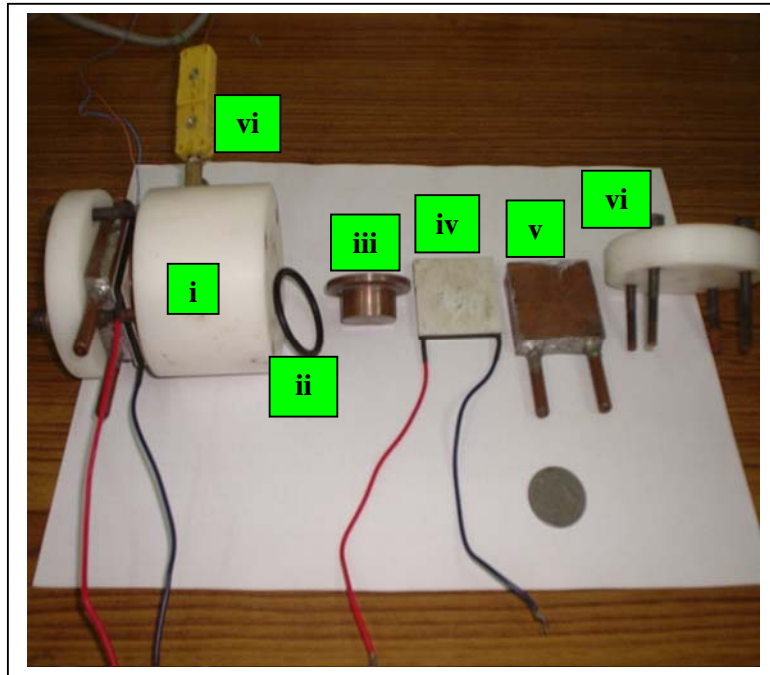


Figure 5.4 Photograph of disassembled test cell:(i) cylindrical block (POM), (ii) 'O'ring, (iii) removable disk (copper), (iv) Peltier element, (v) heat sink (copper), (vi) cover disk (POM), (vii) K-type thermocouple.

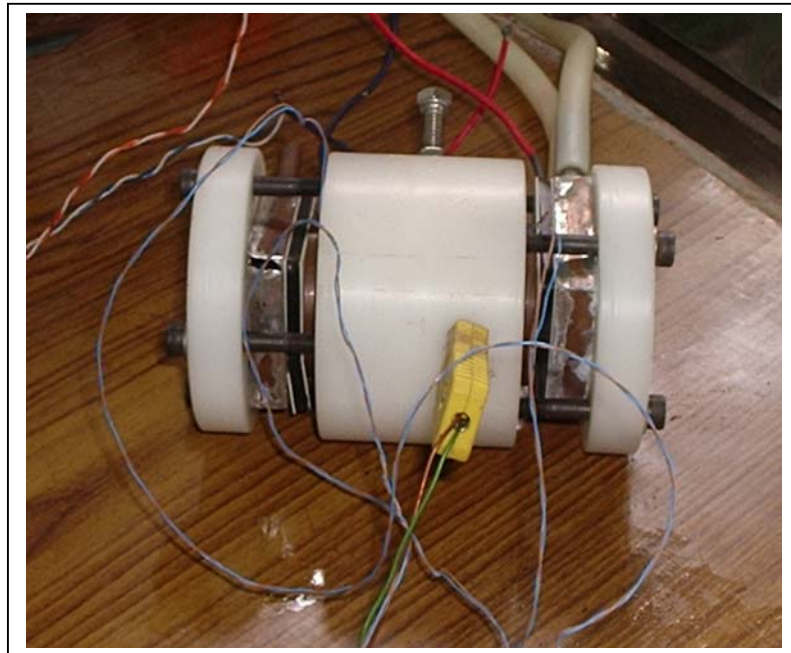


Figure 5.5 Photograph of assembled test cell.



Figure 5.6 Photograph of thermostatic bath.



Figure 5.7 Photograph of data acquisition system (ADAM module 4018 and converter 4520).

5.2.3 Power Supply System and Signal Generator

The line voltage, 230V A.C., 50Hz is stabilized (servo voltage stabilizer) before feeding to different equipment/instruments. The signal generator generates the oscillating signal (0-5V) of required frequency. The time period of this signal can be varied from 20s to 400s depending on the requirement. If two different signals are required, two channels signal generator with phase shifter has to be incorporated. The D.C. (0-15V) power supply is used to modulate the oscillating signal over the D.C. voltage. The role of the amplifier is to regulate the amplitude of the oscillating part and also regulates the current rating (0.5-4A) for the Peltier element. The regulation of D.C. power source regulates the mean temperature of the Peltier element whose other surface is maintained at constant temperatures. This type of two-frequency signal generator is not available commercially. For this experiment it has been specially fabricated. However, in semi-infinite samples one signal generator is adopted.

5.2.4 Data Acquisition System

The temperature at the input and output of the sample are measured by K-type thermocouple probe (0.5 mm). These thermocouples are connected to a PC through ADAM 4000 series data acquisition modules as shown in Fig. (5.7). These data acquisition modules consist of ADAM 4018 (8-channel) thermocouple input module and ADAM 4520 (isolated RS-232 to RS-422/485) converter. The converter is connected to the COM port of the PC. The temperature data collected from the two channels are stored in computer for further analysis.

5.3 Experimental Procedure

At the beginning of experiment the distance between thermocouples are checked accurately. Then the test section is filled with liquid through a hole provided in middle of the cell. The liquid used in experiments are ethylene glycol, ethanol, glycerol and water. The temperature of copper disc is given periodic oscillation by Peltier element at one side of the sample with a constant frequency. The cooling chamber is supplied with water from a constant temperature bath, which act as a partial controller of the mean temperature of oscillation. The

temperature oscillation in this element is further controlled by adjusting the D.C. component and oscillating component to obtain two objectives:

- a. The oscillation amplitude is adjusted to be kept small enough (of the order of 2K) within the test liquid to retain constant fluid properties and to avoid natural convection in the liquid. The amplitude is not allowed to decrease too much to affect the accuracy of the measurement.
- b. The smaller amplitude and accurate adjustment of the mean temperature of oscillation by controlling D. C. component ensure that the test is carried out at the required temperature for the sample.

The measurements of temperatures are made at different positions; at the interface of the copper disc and on the sample, 5.0 mm from the first thermocouple, and at other end of the sample similar to the first location. For this purpose two K-type thermocouples of 0.2 mm diameter are used at the interfaces of the copper disc and the liquid sample and one 0.5 mm diameter K-type thermocouple probe. Response of the thermocouples is connected to data acquisition system. This data acquisition system is connected to a PC for data recording and analysis. The initial oscillations are not used for computation because of their transient nature. After the equilibrium condition has been reached, temperature oscillation is recorded for a number of cycles. Thermocouples attached in the interface between far end and liquid sample assures that the oscillations are completely died down and thus the approximation of semi-infinite medium holds true. It may be noted that spacing between the two thermocouples may be adjusted depending on the sample to get a semi-infinite medium.

The thermocouple at the 5 mm inside the specimen is used to collect the output response for the input response at one end of the sample. By evaluating amplitude or phase of the temperature oscillation at the two points, the thermal diffusivity can be determined. In the present experiment the amplitude ratio is considered to measure the thermal diffusivity. The preassigned data for the experiments are tabulated in Table-5.1.

5.4 Data Reduction and Analysis

Different liquid samples such as, ethylene glycol, ethanol, glycerol and water are taken for the measurement of thermal diffusivity. The temperature oscillation

data directly recorded by the data acquisition system at two points (i.e., input and output) during experiment are analyzed by FFT to evaluate the fundamental amplitude or phase. Typical temperature responses of the two thermocouples and its corresponding FFT results are plotted in Figs. (5.8) to (5.11) for the four samples. Test results are provided in Table-5.1. The results include measurements of spacing between thermocouples, x , periodic time, t_p , and average mean temperature of oscillation. The spacing between two thermocouples has been calculated by a Vernier caliper and the time period has been estimated from the signal generator. The mean temperature of oscillation has been estimated from the D.C. components of FFT analysis, which has been explained below. Measurement data of each sample is presented in tabular form, Table-5.2 to 5.5. The steps of the calculation procedure for the evaluation of amplitude and phase values have been shown by sample calculations.

The FFT of any periodic signal gives complex quantities (C_n ; $n = 0, 1, \dots$). Thus the complex quantity of first input temperature signal in ethylene glycol is,

$$C_0 = 32.97379; \quad 000000 \quad (5.2)$$

$$C_1 = 0.666852; \quad 0.245693 \quad (5.3)$$

The first coefficient with zero suffixes is the D.C. component. The second coefficient with suffix one consists of real and imaginary parts respectively. The Fourier coefficients a_1 and b_1 can be calculated from Equations (4.39) and (4.40) to give

$$a_1 = 1.333704 \quad \text{and} \quad b_1 = -0.491386 \quad (5.4)$$

By substituting the values of a_1 and b_1 in Equations (4.33) and (4.34), the amplitude and phase are obtained as,

$$\text{Amp.1} = A_1 = [(1.333704)^2 + (-0.491386)^2]^{0.5} = 1.4213 \quad (5.5)$$

$$\text{and phase } (\Phi_1) = \tan^{-1} (-0.491386/1.333704) = -0.357 \text{ radian} \quad (5.6)$$

In the similar way, input and output amplitudes are calculated for each cycle and every liquid samples. By taking the ratio of output to input amplitudes results amplitude ratio. Once the amplitude ratio ($\text{Amp.2}/\text{Amp.1}$) has been obtained. The thermal diffusivity (α) of the sample is calculated by using Eq. (4.20). By substituting the corresponding experimental values $x = 5.5 \text{ mm}$, $t_p = 241\text{s}$ and amplitude ratio $A = 0.1217$ into Eq. (4.20) the thermal diffusivity is calculated to be,

$$\alpha = 8.8905 \text{ m}^2/\text{s} \quad (5.7)$$

The calculated results of input amplitude (Amp.1), output amplitude (Amp.2), amplitude ratio, and thermal diffusivity are tabulated in Table-5.2 to Table-5.5 for each sample. The mean value of amp.1 and amp.2 are given at the end of the table. Also mean and standard deviation of thermal diffusivity and amplitude ratio are calculated using Eq. (4.45). All these values are presented at the end of the table for each sample.

Table 5.1: Preassigned data for the experiment.

Liquid Sample	Spacing between thermocouple (mm)	Periodic time (s)	Mean temperature of oscillation ($^{\circ}\text{C}$)
Ethylene glycol	5.5	241	32.6
Ethanol	5.5	203	32.2
Glycerol	4.4	203	32.9
Water	5.5	204	35.8

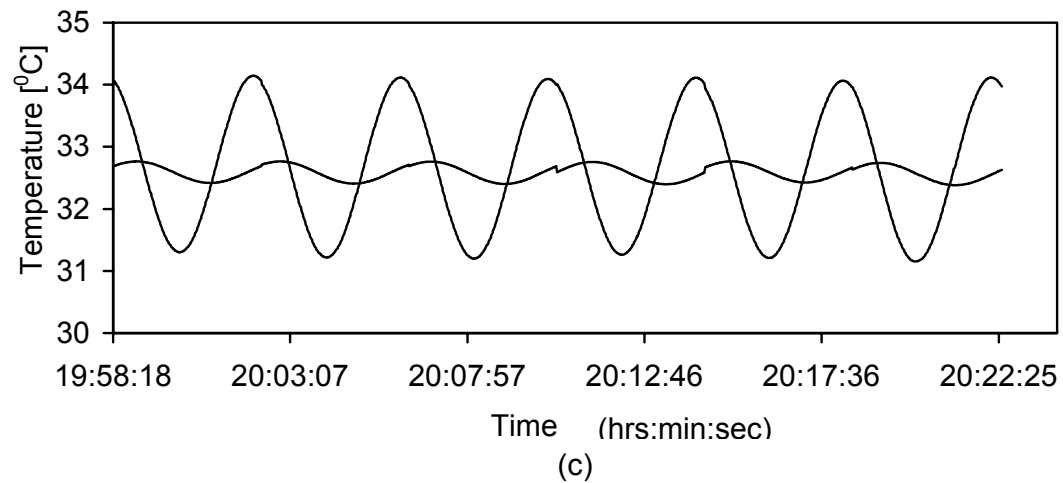
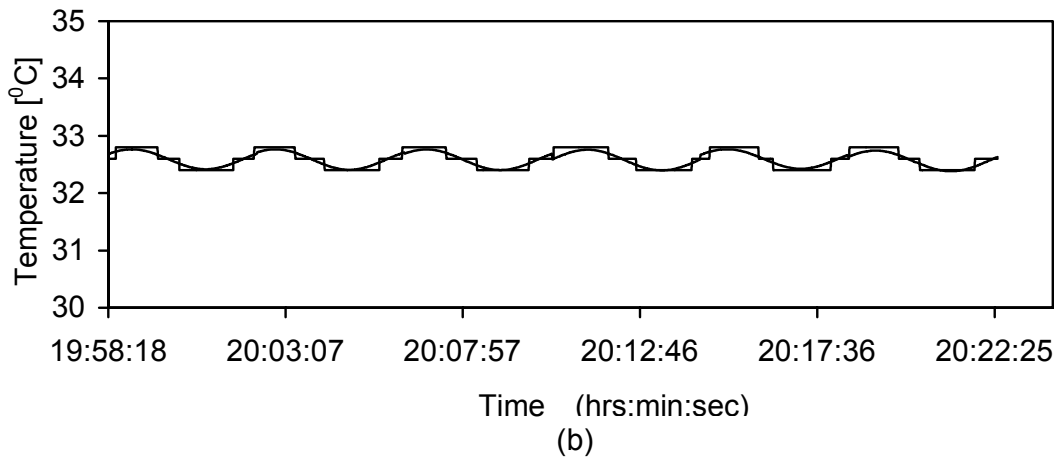
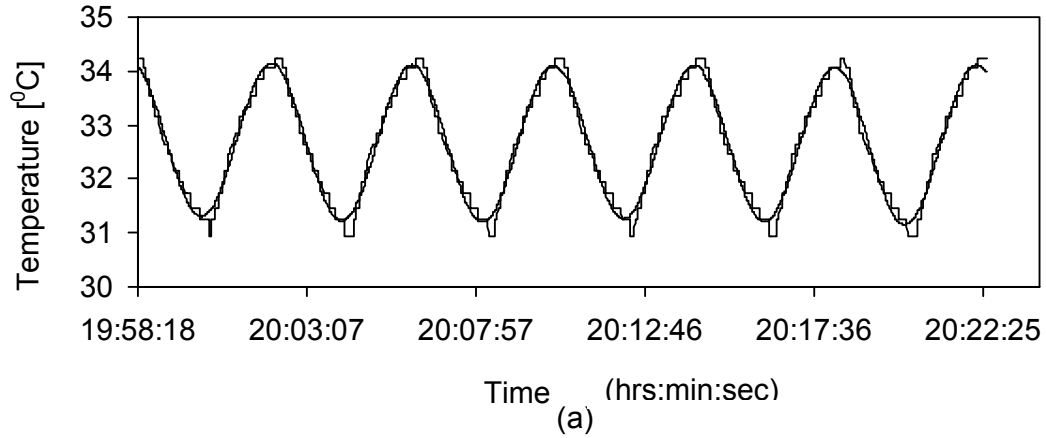
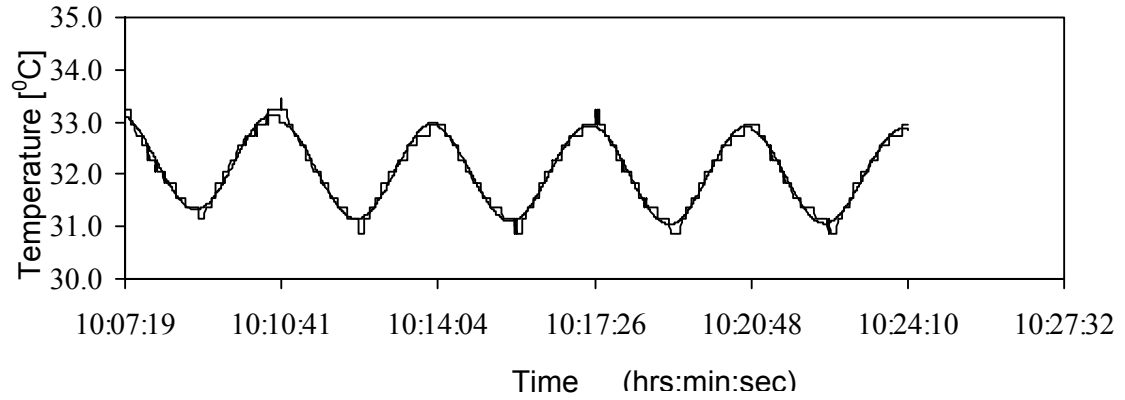
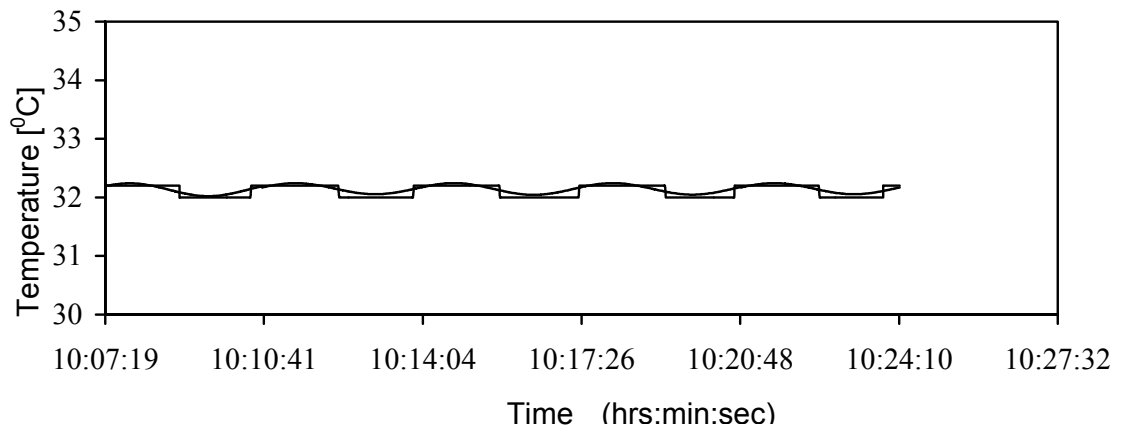


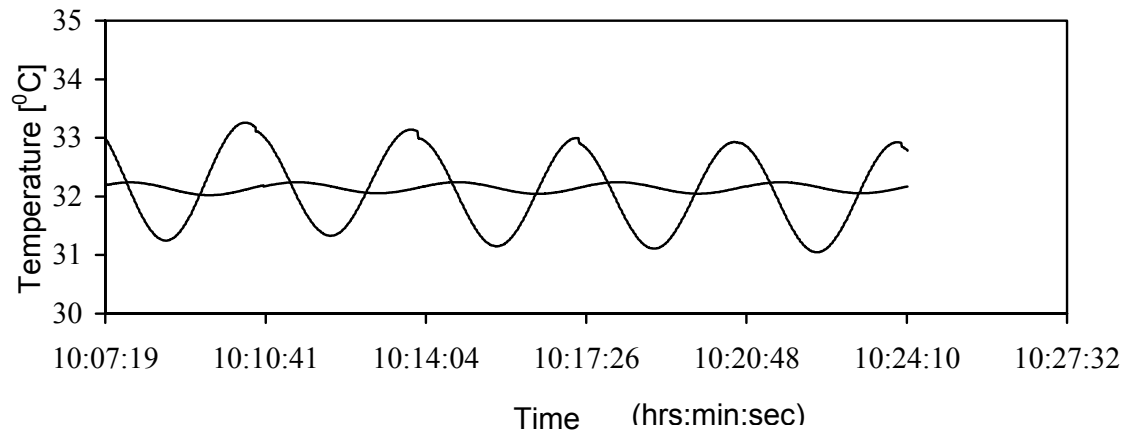
Figure 5.8 Thermocouple response of ethylene glycol at two points for (a) input temperature oscillation and its FFT, (b) output temperature oscillation and its FFT, (c) FFT of input output signals.



(a)



(b)



(c)

Figure 5.9 Thermocouple response of ethanol at two points for (a) input temperature oscillation and its FFT, (b) output temperature oscillation and its FFT, (c) FFT of input output signals.

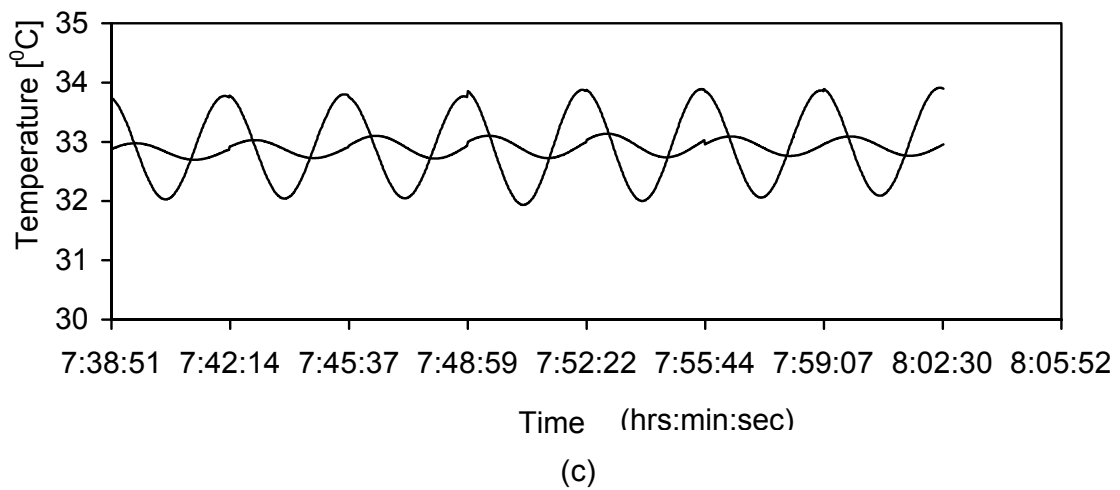
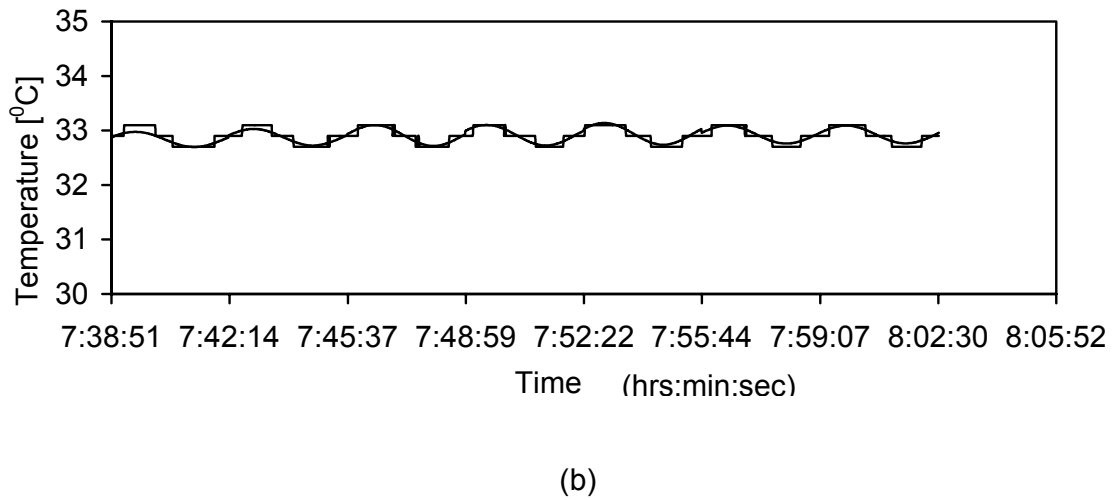
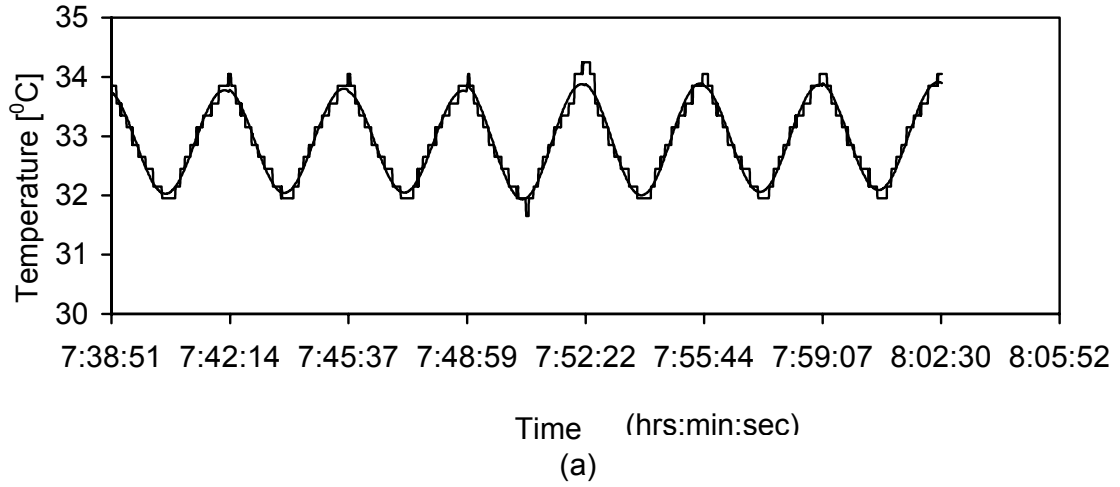
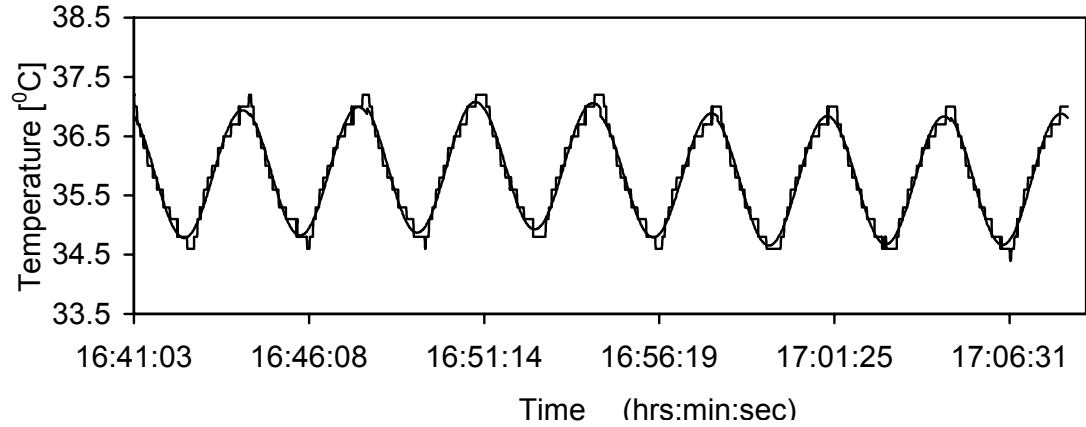
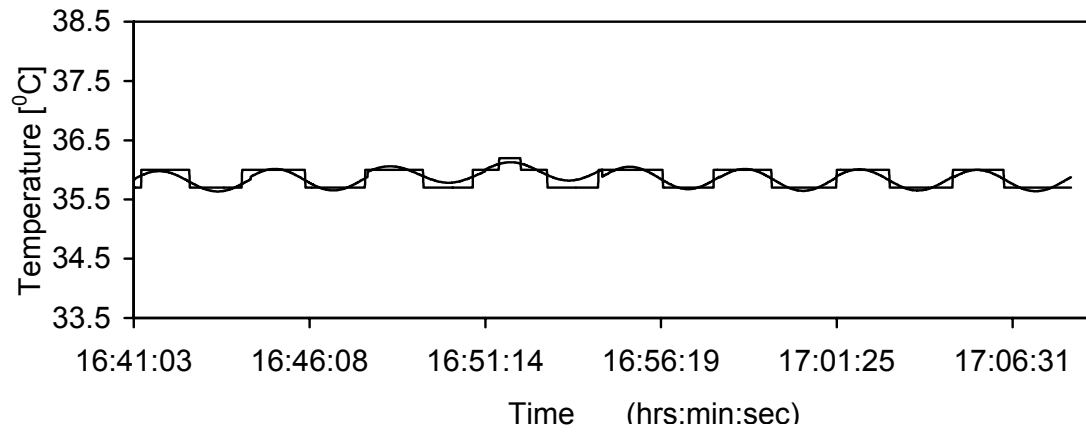


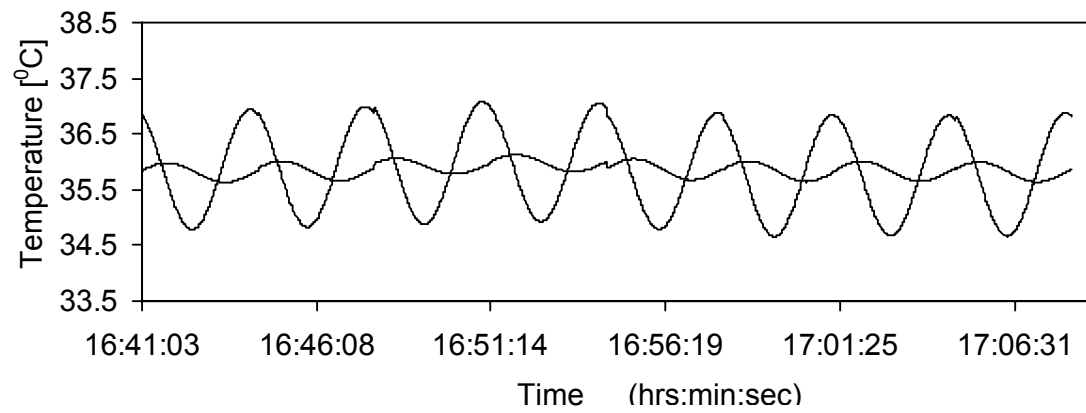
Figure 5.10 Thermocouple response in glycerol at two points for (a) input temperature oscillation and its FFT, (b) output temperature oscillation and its FFT, (c) FFT of input output signals.



(a)



(b)



(c)

Figure 5.11 Thermocouple response of water at two points for (a) input temperature oscillation and its FFT, (b) output temperature oscillation and its FFT, (c) FFT of input output signals.

Table 5.2: **Measurement data and the calculation of thermal diffusivity for ethylene glycol.**

Sl. No.	Amp.1	Amp.2	Amp.2/Amp.1	Diffusivity, E-08 m ² /s
1	1.4213	0.1730	0.1217	8.8905
2	1.4484	0.1780	0.1229	8.9722
3	1.4463	0.1796	0.1242	9.0618
4	1.4249	0.1798	0.1262	9.2026
5	1.4273	0.1700	0.1191	8.7100
6	1.4813	0.1799	0.1214	8.8716

Average Amp.1: 1.4416

Average Amp.2: 0.1767

Thermal diffusivity: $8.9515\text{E-}08 \pm 0.0692\text{E-}08 \text{ m}^2/\text{s}$.

Amplitude ratio: 0.1226 ± 0.000996

Table 5.3: **Measurement data and the calculation of thermal diffusivity for ethanol.**

Sl. No.	Amp.1	Amp.2	Amp.2/Amp.1	Diffusivity, E-08 m ² /s
1	0.9065	0.1089	0.1201	10.4244
2	0.9246	0.0937	0.1013	08.9328
3	0.9095	0.0997	0.1096	09.5787
4	0.9393	0.0978	0.1041	09.1477
5	0.9076	0.0937	0.1032	09.0794

Average Amp.1: 0.9175

Average Amp.2: 0.0988

Thermal diffusivity: $9.4326\text{E-}08 \pm 0.2702\text{E-}08 \text{ m}^2/\text{s}$.

Amplitude ratio: 0.1077 ± 0.003401

Table 5.4: **Measurement data and the calculation of thermal diffusivity for glycerol.**

Sl. No.	Amp.1	Amp.2	Amp.2/Amp.1	Diffusivity, E-08 m ² /s
1	0.8737	0.1397	0.1599	08.9150
2	0.8798	0.1514	0.1721	09.6749
3	0.9705	0.1884	0.1941	11.1499
4	0.9438	0.1992	0.2111	12.3811
5	0.9040	0.1651	0.1826	10.3638
6	0.9107	0.1638	0.1799	10.1799

Average Amp.1: 0.9138

Average Amp.2: 0.1679

Thermal diffusivity: $10.4441\text{E-}08 \pm 0.491812\text{E-}08 \text{ m}^2/\text{s}$.

Amplitude ratio: 0.1833 ± 0.007243

Table 5.5: **Measurement data and the calculation of thermal diffusivity for water.**

Sl. No.	Amp.1	Amp.2	Amp.2/Amp.1	Diffusivity, E-08 m ² /s
1	1.0777	0.1715	0.1591	13.7897
2	1.0845	0.1809	0.1668	14.5241
3	1.0465	0.1881	0.1797	15.8159
4	1.0916	0.1843	0.1688	14.7222
5	1.0790	0.1786	0.1655	14.3998
6	1.1075	0.1797	0.1623	14.0859

Average Amp.1: 1.0811

Average Amp.2: 0.1805

Thermal diffusivity: $14.5562\text{E-}08 \pm 0.285705\text{E-}08 \text{ m}^2/\text{s}$.

Amplitude ratio: 0.1670 ± 0.0029

5.5 Error Estimation

Uncertainty is an estimate of experimental error. The total uncertainty or the total error is the sum of the norms of bias error and precision error. A detail description of the theory of error analysis is given in Chapter 4.4. In this section, the error in measurement of thermal diffusivity has been estimated from the measured data for the four samples, tabulated in Table-5.1 to 5.5. The total uncertainty is given in Table-5.6. The calculation procedure for this total uncertainty is outlined by an example. The calculations on ethylene glycol serves as the example.

The precision error is attributed by three variable; distance, time period and amplitude ratio. For the same sample holder and same wave generator, the contributions of precision error due to these variables are zero. Thus precision error is controlled by the amplitude ratio alone. This error evaluated from Eq. (4.63) is,

$$S_r = \frac{\delta\alpha}{\alpha} = \left[\left(\frac{2\delta A}{A \log_e A} \right)^2 \right]^{0.5} \quad (5.8)$$

The value of A is the average value of amplitude ratio (0.1226). The random error δA (0.000996) is calculated from Eq. (4.48). The precision limit in the result becomes

$$\begin{aligned} S_r &= \frac{\delta\alpha}{\alpha} = \left[\left(\frac{2(0.000996)}{0.1226 \log_e (0.1226)} \right)^2 \right]^{0.5} \\ &= 0.77\% \end{aligned} \quad (5.9)$$

This value is tabulated in Table-5.6 under the heading random limit for ethylene glycol. For the total number of readings, $N = 6$, the number of degrees of freedom ν ($= N - 1$) is 5. Using this value of ν , t-distribution table gives,

$$t_d = 2.571 \quad (5.10)$$

Thus the total contribution of precision error is $t_d S_r$.

The contribution of bias errors are attributed due to measurement of spacing between two thermocouple, time period, and accuracy of thermocouple. The spacing between thermocouple is measured by Vernier caliper whose least count is 0.1 mm. The period time of a cycle is measured in the scale of seconds, where the bias error contribution is 1 second. The thermocouples are K-type. As per manufacturing specification the error is 0.0077 at 285°C. Considering an average

value over the temperature range, the bias error of 0.01K is widely accepted. These values are tabulated in the Table-5.6.

Using Equations (4.63) and (4.64)

$$B_r = \frac{\delta\alpha}{\alpha} = \left[\left(\frac{2(0.1)}{5.5} \right)^2 + \left(\frac{1}{241} \right)^2 + \left(\frac{2\delta A}{A \log_e(0.1226)} \right)^2 \right]^{0.5} \quad (5.11a)$$

$$\text{and } \frac{\delta A}{A} = \sqrt{\left(\frac{0.01}{0.1767} \right)^2 + \left(\frac{0.01}{1.4416} \right)^2} = 0.057011348 \quad (5.11b)$$

It may be noted that in the Eq. (4.64) the percentage error in output and input temperatures refer to the percentage error in amp.1 and amp.2 respectively. Thus the bias limit of thermal diffusivity for ethylene glycol is obtained as,

$$B_r = \frac{\delta\alpha}{\alpha} = \left[\left(\frac{2(0.1)}{5.5} \right)^2 + \left(\frac{1}{241} \right)^2 + \left(\frac{2(0.057011348)}{\log_e(0.1225)} \right)^2 \right]^{0.5} \\ = 6.55\% \quad (5.12)$$

The total uncertainty in thermal diffusivity with 95% coverage is determined from Eq. (4.59). Hence using the numerical values from Eqs. (5.9), (5.10) and (5.12), the total uncertainty is estimated as,

$$U_r = \left[(6.55)^2 + ((2.571)(0.77))^2 \right]^{0.5} \\ = 6.85\% \quad (5.13)$$

The total uncertainty of different liquid samples is tabulated in Table-5.6. The measured value of thermal diffusivity and reported literature value are tabulated in Table-5.7 for comparison.

$$\% \text{ Deviation} = \frac{(\text{standard value} - \text{measured value})}{\text{standard value}} \times 100 \quad (5.14)$$

Using the standard value and measured value of thermal diffusivity of ethylene glycol, the % deviation becomes

$$\% \text{ Deviation} = \frac{(9.56 - 8.95)}{8.95} \times 100 = 6.38\% \quad (5.15)$$

The calculated total uncertainty assessment is used for plotting the error bar in the measurement of thermal diffusivity. The figure for error bar is shown in Fig. (5.12). This figure shows that the reported standard values of thermal diffusivity are well within experimental uncertainty of the present experiment.

Table 5.6: **Experimental uncertainty in thermal diffusivity measurement.**

Liquid sample	Bias error			Random error in amplitude ratio ($\delta A/A$) %	Uncertainty in diffusivity		
	Spacing between thermocouples (mm) (δx)	Periodic Time (s) (δt_p)	Temp. Amps. (K) (δT_{out}), (δT_{in})		Bias limit (B_r) %	Random limit (S_r) %	Total (U_r) % $U_r = [B_r^2 + (t_d S_r)^2]^{0.5}$
Ethylene glycol	0.1	1	0.01	0.81	6.55	0.77	06.85
Ethanol	0.1	1	0.01	3.16	9.85	2.83	12.61
Glycerol	0.1	1	0.01	3.95	8.48	4.66	14.67
Water	0.1	1	0.01	1.74	7.27	1.94	08.82

Table 5.7: **Comparison of measured thermal diffusivity of liquid samples.**

Sample material	Measured value of thermal diffusivity E-08 m ² /s	Reported value E-08 m ² /s	Deviation from [153]
Ethylene glycol	8.95 ± 6.85%(32.6 ⁰ C)	9.56(32.6 ⁰ C) Perry et al. [153] 9.19(24 ⁰ C) Baladers et al. [116]	6.38 %
Ethanol	9.43 ± 12.61%(32.2 ⁰ C)	8.59 (32.2 ⁰ C) Perry et al. [153] 9.323 (20 ⁰ C) Czarnetzki et al. [8] 8.35(23.5 ⁰ C) Sun et al. [110]	9.77 %
Glycerol	10.44 ± 14.67%(32.9 ⁰ C)	9.42 (32.9 ⁰ C) Perry et al. [153] 9.705(20 ⁰ C) Czarnetzki et al. [8] 9.40(23.3 ⁰ C) Sun et al. [110] 10.0(17.7 ⁰ C) Fontana et al. [146]	10.82 %
Water	14.55 ± 8.82%(35.8 ⁰ C)	14.96 (35.8 ⁰ C) Perry et al. [153] 14.12 (20 ⁰ C) Czarnetzki et al. [8] 14.23(24 ⁰ C) Baladers et al. [116] 14.0(17.6 ⁰ C) Fontana et al. [146]	2.74 %

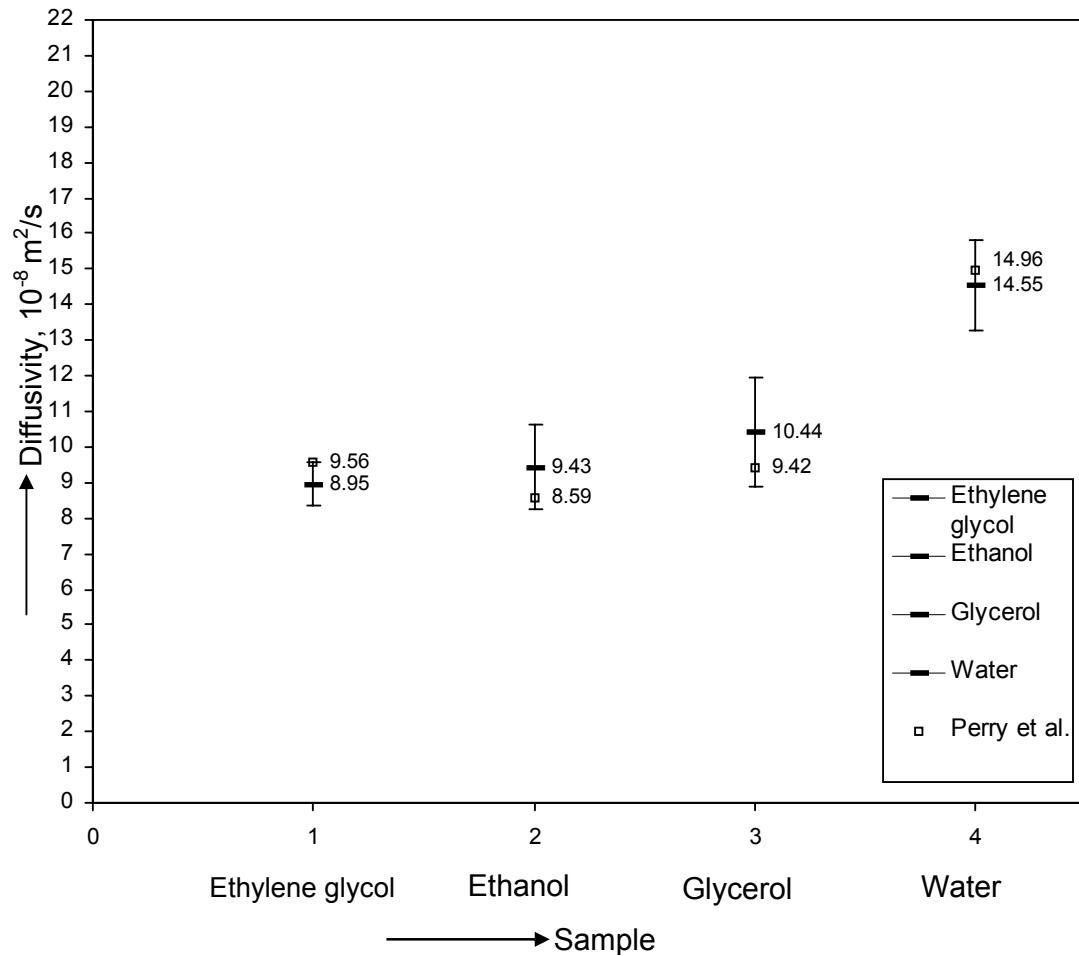


Figure 5.12 Comparison between reported and present values of thermal diffusivity of liquid samples.

5.6 Conclusions

The generation of sinusoidal temperature wave and accurate measurement of amplitude and phase shift have been a problem in the past. In earlier, the temperature oscillations are established by oscillating the heat flux by an electrical resistance heating. A disadvantage of the resistance heater is that the time average of the heat supplied during each cycle cannot be equal to zero. This leads to an increase in the mean temperature of the bar during each cycle, which precludes the measurements at the initially specified temperature. In comparison to this, the

Peltier element operated by modulated signal proves to be stable, reproducible, and easy to control. However, the amplification of such a low frequency oscillating signal for higher current rating (3A) is critical.

In the measurement of liquid samples, in the present experiment, the amplitude of the temperature wave falls off rapidly at a short distance from the heated end. This requires an adequate spacing of the thermocouple for the amplitude measurement of output signal. On the other hand very short spacing results an inaccurate measurement of phase shift. Thus an appropriate spacing has been adopted based on the Eq. (5.1) to measure amplitude of the temperature wave. Also it has been observed that thermal diffusivity is more sensitive to amplitude attenuation. Thus, the amplitude information is used to get the experimental results.

The random error associated to the four samples is small as compared to the bias error. This is one of the advantages of temperature oscillation method. During every period, the whole cycle of changes is repeated. This reduces the random error and increase the signal to noise ratio.

The Table-5.7, presents the reliability of such experiment by comparing the present result with other reported values in the literature. The comparison shows an excellent agreement, as it is evident from the Fig. (5.12). Thus the simplicity in experimental procedure based on a lucid theory of temperature oscillation, establishes an inexpensive measurement technique, which can lead to achieve reliable results.

In general, the principle of temperature oscillation is restricted during first order phase change. For a class of limited specialized materials, the very temperature oscillation brings permanent changes in thermophysical properties of materials due to phase change. It is very common in 2nd order phase transitions such as super conductor to normal conductor, ferromagnetism to Para magnetism etc. for these types of materials the temperature oscillation has its own limitations. However for commonly used engineering materials, this limitation is not required.

Chapter 6

Conclusions and Scope for Future Work

Over the years, a number of methods have been developed to measure thermal transport properties of many different materials. To keep up with the fast development of new materials and the increasing importance of accommodating new applications, accurate and reliability of experimental data are essential. Also the use of short measuring time and prediction of a set of thermophysical properties from a single experiment are some of the distinctive features of an efficient experimental procedure. The novel transient technique based on the temperature oscillation method has been adopted in the present work for the measurement of thermophysical properties. The temperature oscillation method is a pseudo-steady state oscillation where it combines the advantages of a steady state measurement with the potential to measure a property describing a non-steady state. Short measuring time and prediction of a set of thermophysical properties from a single experiment are the inheritant properties of temperature oscillation method.

6.1 Conclusions

The thesis has been organized to give detailed conclusions for each aspect of the work in their respective chapters. Therefore, this section provides a brief summary of the work and the important conclusions. A detailed literature survey

has been conducted in Chapter 2 to summarize the different methods including the currently used methods for the determination of thermophysical properties.

A generalized solution based on Laplace transform has been derived in chapter 3. For this case, sinusoidal temperature oscillation on both sides of finite sample with constant angular frequency, but with different amplitudes and phases are considered. The general solution has been reduced to different boundary conditions available in the literature. Expressions of temperature distribution in a finite length of the sample for different possible practical situations have also been presented. To illustrate the settling time for transients to settle down, the expression for settling time has been derived and also been presented in graphical form.

The mathematical model on semi-infinite medium based on Laplace transform for the present experimental model has been presented in Chapter 4. This chapter includes the theory of error analysis and also for the sake of mathematical completeness, settling time has been derived. In the preceding chapter the experimental setup along with the experimental procedure have been described. The oscillating heat source based on Peltier element, excited by D.C. voltage modulated square wave signal, seems to be the novel method to determine the thermal diffusivity. The measurement of amplitude attenuation for the fundamental frequency is more effective for the determination of thermal diffusivity rather than the measurement of phase change. Due to the peculiar characteristics of liquid, four liquid samples ethylene glycol, ethanol, glycerol and water are selected to validate the experimental procedure. The result shows an excellent agreement with the reported values in literature. Due to the periodic nature of temperature oscillation method, the small contribution of random error leads to yield a low value of total uncertainty in measurement. Thus based on the lucid theory, the simplicity in experimental procedure establishes an inexpensive measurement technique.

6.2 Scope for Future Work

- The experimentation on semi-infinite medium can be extended to finite samples with the presence of oscillating heat sources at both the ends of the sample.

- Temperature oscillation technique can be extended to pressurized gases to measure their thermophysical properties.
- Using the Infrared technique to measure the temperature, the present technique can be extended to measure the thermophysical properties of the samples more accurately.
- In the hyperbolic heat conduction, the temperature oscillation method has the potential to measure the relaxation time.

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